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Title: Integrated Research & Training in Space-Molecular Biology

Preliminary Report on Participation in Luster Project

Institution: The University of Chicago

Principal Investigator: Humberto Fernandez-Moran, M.D., Ph.D.
Professor of Biophysics
Department of Biophysics

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SUMMARY

Specimens collected by the Luster Sounding Rocket Experiment during the Leonid meteor shower November 16, 1965 were examined by combined high resolution electron microscopy and electron diffraction techniques. As participants in the guest scientist program and in close collaboration with Neil H. Farlow of the Space Sciences Division, Ames Research Center, NASA, we were able to share part of one Ames module, which was handled at Ames Research Laboratory, conforming to the rigorous standards of the contamination control program which was applied to all phases of the experiment. The Luster micrometeoroid sampling instrument was carried by an Aerobee 150 sounding rocket to an altitude of 145 km. The instrument opened, on ascent, at 64 km and closed, on descent, at 116 km, exposing 1 square meter of sampling surface vertically and another square meter horizontally for 200 seconds.

Special high vacuum containers were designed and constructed for transfer of the sampling surfaces under controlled conditions of minimum contamination. Each high vacuum container provides secure attachment for demountable slides containing several hundred platinum specimen holders coated with extremely smooth, thin (100Å-400Å) film substrates of carbon-silicon oxide-formvar, in addition to larger surfaces of freshly cleaved mica, and plastic slides. Three identical containers and specimen substrate assemblies were prepared for the Luster experiments to furnish independent contamination controls at the different sites of the experiment.

Contamination control was maintained throughout the transportation, flight, recovery, and analysis of the sampling surfaces. The specimens were examined in the new electron microscope facility in the Research Institutes of the University of Chicago. These laboratories, equipped with five high resolution electron microscopes and diffraction cameras, were especially designed to operate under clean room conditions. They have a special air-conditioning system provided with Cambridge absolute filters excluding particles greater than 0.1 microns.

All studies were performed under conditions of minimized specimen contamination and radiation damage by using electron microbeam illumination of very low intensity and liquid nitrogen cooling devices. This represents a significant improvement over previous studies, since hereby the appreciable specimen heating and contamination effects, frequently occurring in standard electron microscopy, could be considerably reduced, thus making it possible to detect certain electron sensitive materials for the first time. In addition, the special liquid nitrogen traps surrounding the specimen practically eliminate the serious additional contamination from organic vapors in the microscope column, thereby enabling us to consistently detect any particles in the size range of 10Å to 20Å collected on the atomically smooth carbon film and single-crystal surfaces.

The improved resolution in the range of 10Å to 20Å reproducibly achieved in these studies effectively enlarges the range of direct

visualization, not only of significant new specimen details, but also of many types of hitherto undetected contaminants.

In keeping with the rigorous criteria for identification of particle origin described by Farlow, Blanchard, and Ferry, great emphasis was placed on thorough evaluation of possible sources of contamination, with particular attention to systematic examination of control specimens.

The results described in this report are based on careful examination of several thousand specimen areas, and evaluation of representative electron micrographs recorded at electron optical magnifications of 250x to 180,000x, including the corresponding selected area high resolution electron diffraction patterns. This preliminary survey, carried out by two independent electron microscope groups working with different instruments, represents examination of only 20% of the platinum specimen holders exposed during the Luster Flight, and of the corresponding non-flight controls, and less than 10% of the total available sampling area in our experiment.

Although much further work remains to be done before final evaluation of the results can be attempted we believe that certain reproducible findings made in the course of this preliminary investigation are of significance, and appear to confirm and extend the results of previous work in this field.

As shown in the accompanying illustrations a number of characteristic particles have been found in the Luster on-flight specimens which are essentially similar to the three types of micrometeorite particles first described by Hemenway and Soberman (Astronomical Journal, 67, 256, 1962). In agreement with the findings of these authors, most of the particles encountered are submicron in size (about 0.01μ to 0.4μ). However, with improved resolution we were able to detect numerous particles measuring only about 20Å to 100Å, and to find indications of subunit structure in the larger particles, which also yield characteristic electron diffraction patterns.

Application of low intensity electron microbeam illumination and specimen cooling permitted us to make the unexpected finding of an opaque electron-sensitive material which appears to coat many of the particles and is volatilized or degraded by electron bombardment. High resolution electron diffraction patterns with long period spacings recorded from this material have features in common with certain organic compounds which are being further investigated. Although this electron-sensitive material has not yet been found in non-flight controls, considerable skepticism concerning its extraterrestrial origin is indicated until all possible sources of contamination can be eliminated. If subsequent studies with improved methodology substantiate the existence of such volatile surface layers associated with certain types of meteor stream particles, it is interesting to speculate on the possibility that we may be dealing with characteristic organic compounds, or with (mixed) gas hydrates of organic compounds.

Collateral electron microscopic and electron diffraction studies are being performed on samples of Canyon Diablo meteorties (kindly provided by Dr. E. Anders) and on other known samples of representative iron and stony meteorites. Examination of this material will provide a valuable reference standard designed to test and refine our techniques, and to otherwise supplement the current electron-optical investigations of micrometeoroid material collected by rocket payload.

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I. Specific Research Program.

A. Report on Preliminary Examination by Electron Microscopy and Electron Diffraction of Materials Collected by the Luster Sounding Rocket Experiment on November 16, 1965.

INTRODUCTION

Following the invitation of Dr. Maurice Dubin, Chief of Interplanetary Dust, Physics and Astronomy Program, OSSA of NASA, and in close collaboration with Dr. Neil H. Farlow of the NASA Ames Research Center, we were able to participate in the Luster Rocket Collection Experiment, successfully carried out on November 16, 1965.

LUSTER is a sounding rocket program designed to collect meteor stream particles and other extraterrestrial dust from the outer fringe of the earth's atmosphere for laboratory analysis. Some of the material, to be gathered in the course of the planned series of three collections, is presumed to be of lunar origin (D. E. Gault et al, NASA TN D-1767, 1962). The experimental objective of the 1965 Luster Flight was to collect micrometeoroid debris during the peak of the Leonid meteor shower.

The Leonids is an annually recurring meteor shower which has attracted considerable attention, with records of its appearance dating back to 585 A.D. (W.K. Green, 1947). It is attributed to the comet Tempel-Tuttle (1866I) whose orbit coincides with that of the meteor stream (J. G. Porter, 1963). There is a strong condensation of the meteors into a swarm through which the earth has been passing every 33 or 34 years. Probably the most striking display occurred in November, 1833 when 650 meteors were counted by one observer during fifteen minutes. In 1899 Jupiter passed very close to the meteor swarm and deflected it from the earth's orbit. Subsequent perturbations further decreased its intensity so that in 1932-1934 no real showers were observed.

On the basis of the 33-year intensity cycle, an increase in the influx rate of particles in the Leonid shower of 1965 or 1966 was predicted. As shown in the figure taken from the recent report on the Luster Experiment by N. H. Farlow, M. B. Blanchard, and G. V. Ferry, radar observations and visual counts of the Leonids during the past years appear to confirm this prediction.

Spectra of the ablation, observed as the Leonid meteor particles enter the earth's atmosphere, indicate that these particles are of the stony type containing the major elements: Ca, Mg, Fe, Si, O, and Na (Rao and Lokanadham, Nature, 197, 169; 198, 77, 1963).

The collection of particles with a recoverable sounding rocket, which was successfully carried out on November 16, 1965 close to the estimated peak of the Leonids, is expected to yield information bearing directly on the physical and chemical features of these meteor stream particles. In addition, we may gain direct access to the original cometary material which is probably the only intact source of particles that condensed from the primordial solar nebula (B. Donn, Ann. New York Acad. Sciences, 119, 5, 1964).

After extensive discussions with Mr. Neil Farlow, Space Sciences Division, Ames Research Center, and following the detailed instructions in the Guide to Guest Experimenters, we proceeded to prepare the sampling slides and transfer containers for participation in the Luster project. Our major concern throughout the planning and implementation of these experiments was to try to conform to the most rigorous requirements for controlled exposure and retrieval of suitable substrates for subsequent high resolution electron microscopy and electron diffraction under conditions of minimum contamination and perturbation (Fernandez-Moran, J. Royal Microscop. Soc., 83, 183, 1964).

The new electron microscope facility in the Research Institutes of the University of Chicago is particularly well suited for this purpose, since it is probably the only existing high resolution electron-optical laboratory especially designed to operate under clean room conditions. These laboratories, equipped with five electron microscopes and electron diffraction cameras, have a special air-conditioning system designed to maintain relative humidity not exceeding 50%, and are provided with Cambridge absolute filters excluding particles greater than 0.1 microns (Figures A, M-1).

Careful study of the pioneering work of C. L. Hemenway, R. K. Soberman and their associates (Astronomical Journal, 67, 256, 1962; Tellus, 16, 84, 1964) was of key value in establishing a standard of reference for investigations in this new field, and in helping us design our experiments.

With the experience gained from the Venus Fly Trap Program, and based on our own extensive studies on specimen contamination, we have taken an approach which appears to be well suited for the proposed collection experiments,

For this purpose, in collaboration with the staff of the Biophysics Department and Central Development Workshop, we have designed and constructed special high vacuum containers for transfer of the sampling surfaces to collect extraterrestrial material for electron microscopic and electron diffraction analysis under controlled conditions of minimum contamination. (Figures 1-6). These containers make it possible to maintain a high vacuum of the order of 10^{-7} to 10^{-8}mm Hg during transport from our laboratories to the clean room area at Ames Research Center, where attachment to the module pans of the rocket-launched payload takes place. The specimens are transported back after exposure under similar high-vacuum conditions for direct examination by electron microscopy, electron diffraction, and related electron optical techniques.

As shown in Figures 3-6, each high vacuum container provides secure attachment for slides containing several hundred platinum specimen holders coated with smooth thin film substrates of carbon-silicon oxide-formvar, in addition to larger surfaces of freshly cleaved mica with atomically smooth surfaces, plastic slides, and related collecting surfaces. The stable, chemically inert, platinum specimen holders with extremely smooth, "noise-free" thin film substrates are more suitable than the standard thin copper mesh electron microscope specimen holders with thin films coated with the more particulate aluminum deposits.

Three identical containers and specimen substrate assemblies were prepared for the Luster project to furnish independent contamination controls at the different sites of the experiment: Ames Research Center, The Rocket

Launching Site at White Sands, and the actual on-flight container for the Luster Flight on November 16, 1965.

The containers were hand-carried to Ames Research Center in the latter part of October and returned to our laboratory in the same fashion shortly after the successful Luster Flight. Thanks to the kind assistance of Neil Farlow, it was possible for us to share part of one Ames module, which was handled at Ames Research Center in a Fed. Std. 209, class 100 laboratory, as described by Farlow and Blanchard in their Luster Flight report. Contamination control was therefore maintained throughout the transportation, flight, recovery, and analysis of the sampling surfaces.

The specimens were examined in our clean room laboratories only a few days after retrieval of the capsule. All studies were performed under conditions of minimized specimen contamination and radiation damage by using electron microbeam illumination of very low intensity and liquid nitrogen cooling devices. This represents a significant improvement over previous studies, since hereby the appreciable specimen heating effects, frequently occurring in standard electron microscopy, could be considerably reduced, thus making it possible to detect certain electron sensitive materials for the first time. Moreover, the special liquid nitrogen traps surrounding the specimen practically eliminate the serious additional contamination from organic vapors in the microscope column, thereby enabling us to consistently detect any particles in the size range of 10Å to 20Å collected on the exceptionally smooth, carbon film surfaces. The improved resolution achieved in the studies effectively enlarges the range of direct visualization, not only of significant new specimen details but also, unfortunately, of many types of hitherto undetected contaminants.

In keeping with the rigorous criteria for identification of particle origin described by Farlow, Blanchard, and Ferry great emphasis was placed on thorough evaluation of possible sources of contamination. Particular attention has been devoted to a systematic examination of representative series of control specimens from the two high vacuum control containers and from parallel laboratory reference specimens. We consider it worthwhile to expend a major effort in compiling what may eventually become an "Archive of submicroscopic contaminants" as an indispensable basis of reference for monitoring the effectiveness of a comprehensive contamination control program.

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The results described in this report are based on careful examination of several thousand specimen areas, and evaluation of representative electron micrographs (recorded at electron optical magnifications of 250x to 80,000x), including the corresponding selected area electron diffraction patterns. This preliminary survey has involved the concerted effort of two independent electron microscope groups, working with different instruments in our laboratory, over a continuous period of six months. However, this study represents examination of only 20% of the platinum specimen holders exposed during the Luster Flight, and of the corresponding non-flight controls, and less than 10% of the total available sampling surface area in our experiment. Obviously, therefore, <u>much further work remains to be done before final evaluation of the results can be attempted.</u>

Bearing in mind these important reservations, we believe that certain findings made in the course of this preliminary investigation are of significance, and appear to confirm and possibly extend the results of previous work in this field.

As shown in the accompanying illustrations (Figs. L1-L18), a number of characteristic particles have been found in the Luster specimens which are essentially similar to the three types of micrometeorite particles first described by Hemenway and Soberman (Astronomical Journal, <u>67</u>, 256, 1962). In agreement with the findings of these authors, most of the particles encountered are submicron in size (about 0.01μ to 0.4μ). However, with improved resolution we were able to detect numerous particles measuring only 50Å to 100Å, and find indications of subunit structure in the larger particles, which also yield characteristic electron diffraction patterns.

Application of low intensity microbeam illumination and specimen cooling permitted us to make the unexpected finding of an opaque electron-sensitive material which appears to coat many of the particles and is volatilized or degraded by electron bombardment (Figs. L7-L15) often leaving a typical halo around the particles. High resolution electron diffraction patterns with long period spacings recorded from this material (Figs. L7-L12) have features in common with certain organic compounds which are being further investigated. Although this electron-sensitive material has not yet been found in non-flight controls, considerable skepticism concerning its extraterrestrial origin is indicated until all possible sources of contamination can be eliminated. Nevertheless, if subsequent studies with improved methodology substantiate the existence of such volatile surface layers associated with certain types of meteor stream particles (e.g. cometary dust), it is interesting to speculate on the possibility that we may be dealing with characteristic organic compounds, or with (mixed) gas hydrates of organic compounds as first suggested by S. Miller (Proc. Nat. Acad. Sci. 47, 1798, 1961).

Finally, collateral electron microscopic and electron diffraction studies are being performed on samples of Canyon Diablo meteorites (kindly provided by Dr. E. Anders) and on other known samples of representative iron and stony meteorites. Examination of this material will provide a valuable reference standard designed to test and refine our techniques, and otherwise supplement the current electron-optical investigations of micrometeoroid material collected by rocket payload.

An important phase of this work is being carried out in close collaboration with Dr. Edward Anders, Dr. R. Hayatsu of the Fermi Institute for Nuclear Studies, University of Chicago, and Dr. M. Studier of the Argonne National Laboratory. These authors have recently found (EFINS 65-115, 1965) that iron and stony meteorites are effective catalysts for a Fisher-Tropsch type reaction between carbon monoxide and hydrogen. In hydrogen-rich mixtures approaching cosmic composition, aliphatic and aromatic hydro-carbons are produced rapidly at temperatures between 25° and 580° C. It would be of interest to establish by electron microscopy and diffraction if there is any similarity with certain electron-sensitive (presumably organic) components found in the collected micrometeoroid specimens. Preliminary experiments along these lines (Figs. M2-8) are described in the following report.

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EXPERIMENTAL METHODS

Flight Sampling Method:

Full details of the rocket payload and sampling instrumentation are given in the comprehensive report by N. H. Farlow, M. B. Blanchard, and G. V. Ferry (Preliminary Examination of Particles collected by the Luster Sounding Rocket Experiment). The Luster micrometeoroid sampling instrument, in which our specimens were included as guest experimenters, was carried by an Aerobee 150 sounding rocket to an altitude of 145 kilometers during the November 15, 1965 Leonid meteor shower. The sampling instrument has a sealed enclosure which rises, exposing the arms when the programmed altitude has been reached (Figs. 1,2). Cleaned and pre-evacuated, sealed modules containing the sampling surfaces were installed on the instrument prior to launching (Fig. 3).

As indicated in the curve depicting time versus altitude data (Figure 3, AAA102-5, Ames Research Center) for the November 1965 Luster Flight, the instrument opened, on ascent, at 64 kilometers, and closed on descent at 116 kilometers, exposing 1 square meter of sampling surface vertically and another square meter horizontally for 200 seconds. The instrument was parachuted to earth after severence from the rocket. The vacuum-sealed modules containing the samples were removed from the instrument and taken to the clean-room laboratory for analysis.

As part of the extensive contamination control program, we had arranged to expose, through the courtesy of Mr. Neil Farlow, one of the special ultrahigh vacuum containers at the Ames Research Center clean room facility, and a second control container at the White Sands facility.

Sampling Surfaces for Electron Microscopy and Electron Diffraction:

As shown in Figures 1-6, three basic types of sampling surfaces for electron microscopy and diffraction were used: (a) Specially designed slides of polished and anodized aluminum, containing several hundred platinum specimen holders coated with thin (100-400A) films of carbon silicon monoxide and carbon on thin formvar substrates, or single-crystal graphite, or mica lamellae (Fernandez-Moran, J. Appl. Phys. 31, 1840, 1960), prepared by a special cleavage technique. This procedure differed from previous experiments, in which copper screens and formvar films coated with aluminum were used. Also, special precautions were taken in our experimental series to use the chemically inert, and mechanically more stable, platinum holders which are thoroughly cleaned by chemical treatment and by heating to incandescence in an ultrahigh vacuum.

Throughout, the smoother and more uniform carbon or silicon monoxide films (Figures C1, C2) were used. The thin film substrates were prepared by evaporation of spectroscopically-pure carbon and/or carbon-silicon monoxide in an ultrahigh vacuum (10^{-8}mm Hg) produced in a Varian unit with an ion pump and a Linde molecular sorption forepump with liquid nitrogen cold trap. Any possibility of contamination with oil vapors was thereby eliminated. The films were stripped off from the freshly cleaved mica on quartz distilled, ultrafiltered water (100Å millipore filters).

Rigorous cleanliness precautions were observed throughout the studies in clean room electron microscope laboratories specially equipped with an air conditioning system, containing Cambridge absolute filters which are expressly designed to exclude particles larger than 0.1μ. The platinum specimen holders of the multiple hole and slit types (Figures C-1), giving an unobstructed slit area of 1 x 0.1mm, were firmly secured in the special holders and could be individually indexed for examination. These platinum holders have proved to be the most reliable and convenient type for subsequent critical high resolution, low temperature electron microscopy.

(b) Larger surfaces of specially selected, freshly cleaved single-crystal mica (mounted on slides of 75 x 25mm). These surfaces are known from previous electron microscopy work to represent the smoothest (atomically smooth) and the cleanest surfaces available, provided that cleavage is carried out under appropriately controlled clean room conditions. In addition, the mica is not only chemically inert, but extremely resistant to heating effects. With these surfaces it is possible to collect not only superficially adhering particles but also particles which have been impacted into the mica layers. There is also the possibility of being able to observe radiation damage and fission tracks by electron microscopy in the single-crystal mica substrates.

(c) Plastic slides of lucite or of epoxy materials (75 x 25mm). The use of these slides was prompted by the interesting observations of C. L. Hemenway and R. K. Soberman (Astronomical Journal, 67, p. 256, 1962). In their report, Hemenway and Soberman indicate that "micrometeoroid particles were found to be sufficiently embedded in the lucite that we were unable to remove them from the substrate." Since most of the embedded particles are larger than a few microns, and hence, impenetrable to the electron beam, it was necessary in their experiments to bombard the micrometeoroid particles with an intense electron beam in order to evaporate the constituent material which then recondenses near the particle.

In order to avoid this destructive mode of analysis, we proposed to prepare serial ultrathin sections (100-200Å thick) of the particles embedded in the lucite by using the special diamond knife and ultramicrotome developed by Fernandez-Moran (Exptl. Cell Res. 5, 255, 1953). Although the plastic slides have been scanned and show indications of impacted particles, in this report we shall concentrate exclusively on a description of the results obtained in the examination of the platinum specimen holders with different types of thin film substrates.

Electron Microscopy and Electron Diffraction:

In contrast to previous experiments, we have taken special precautions to examine the specimens under conditions of minimum specimen damage and contamination. As shown in Figures C1-C2, all electron micrographs and electron diffraction patterns of both representative control specimens and of the Luster Flight specimens were first scanned and photographed at low magnifications (approximately 200x) under conditions of minimized specimen contamination and radiation damage by using electron microbeam illumination of very low intensity (image current density of about 10^{-11} to 10^{-12} ams/sq.cm.) which was obtained with single-crystal pointed filaments in a special gun design developed in our laboratory.

In addition, a liquid nitrogen specimen cold stage was used to serve both as an anticontamination and cooling device for protecting labile specimens from the heating effects of the electron beam. Under these controlled conditions, the contamination rates were reduced to well below .01-0.1Å per second, as compared with rates of contamination of several Å per second and irradiated areas of 10 to 100μ with possible temperature increases locally of several hundred degrees C. in conventional electron microscopes.

All of the electron microscope examinations were carried out by two independent groups working with different instruments. One group (H. Fernandez-Moran) worked with a Siemens Elmiskop IA fitted with specially stabilized power supplies and anticontamination devices. The other group (M. Ohtsuki) used a Hitachi Hu-11A which was likewise fitted with viton gaskets, anticontamination devices, stabilized power supplies, and pointed filament sources.

The electron microscope studies were carried out in a facility which was designed to provide optimum conditions for consistent attainment of high resolution. The microscopes were provided with a highly regulated power supply (50 kilowatt motor generator set, specially designed and manufactured by Westinghouse Co., which is equipped with a new solid state regulator giving better than 0.1% voltage stability and very low harmonic distortion). The microscopes were mounted on vibration free foundations in clean room environments. The electron microscopes were used operating mainly at 80kV, but also at 100kV, 75kV, and 50kV for selected specimens.

The astigmatism of the objective lens was 0.05μ or less. It is important to point out that for all critical work electron micrographs were recorded at electron optical magnifications ranging from 200x to 180,000x on special Kodak high definition 70mm films on a thin polyester base. With this material, it was possible to record up to 90 exposures continuously without breaking the vacuum. This was particularly valuable in recording through-focal series of the delicate specimens and electron diffraction patterns using the cold stages. In addition, Kodak high resolution plates, Ilford N-40 and Geveart Scientia 23B 50 plates were used routinely.

For all critical measurements, calibration of the microscope was carried out at the time of recording the electron micrographs using a diffraction grating replica (cross grating replica of 54,864 lines per inch corresponding to 2,160 lines per millimeter). Additional checks on the calibration were carried out by combined electron microscopy and electron diffraction of selected single-crystal gold and copper pthalocyanin of K_2PtCl_4 crystals.

Measurements were carried out directly on the plates and films using an optical comparator (Nikon Shadowgraph, Model 6C). The plates were developed with Agfa Rodinal, Kodak D72 or D76 under controlled temperature conditions (20°C). The prints were made from the original plates or from contact duplicates taking special precautions to eliminate aberrations, using the Durst S-45 enlarger with point source or with a mercury vapor lamp.

The observations described here are based on the evaluation of more than 1,500 plates and films in which an average resolution of 5 to 20Å was consistently achieved. The high resolution small-angle electron diffraction patterns were taken with special attachments to both types of electron microscopes. The model experiments carried out in collaboration with Dr. Anders and his associates are described separately.

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RESULTS

Electron Microscopy and Diffraction Studies of Control Specimens:

Systematic examinations were made of representative series of control specimens from the two high vacuum control containers and from parallel laboratory reference specimens. The results described are based on careful examination of several thousand specimen areas and evaluation of several hundred representative electron micrographs (recorded at electron optical magnifications of 250x to 180,000x), including the corresponding selected area electron diffraction patterns. This study, however, represents only 20% of the platinum specimen holders exposed during the Luster Flight and of the corresponding non-flight controls, and less than 10% of the total available sampling surface area in our experiment.

Thin films of carbon-silicon-monoxide-coated formvar (ca. 200-400Å) were used to cover the slit aperture of platinum specimen holders (Fig. C-1). These exceptionally stable and chemically inert specimen supports are superior to the standard copper specimen grids. They provide an unobstructed view of the entire slit area which was first scanned and photographed at low magnifications under conditions of minimized specimen contamination and radiation damage by using electron microbeam illumination of very low intensity (image current density of about 10^{-11} to 10^{-12} amps/cm^2), and a liquid nitrogen specimen cold stage.

The film substrates were prepared by evaporation of pure carbon and/or C-SiO in ultrahigh vacuum (10^{-8}mm Hg) of Varian Unit with ion pump and sorption forepump with liquid nitrogen cold trap, thus eliminating any possibility of contamination with oil vapors. The films were mounted on platinum holders (final cleaning was done by heating in a high vacuum) using quartz-distilled, ultra-filtered water and reagents of controlled purity. Each specimen holder was kept in an individual sealed micro-desiccator and provided with separate handling devices to eliminate any contact with the operator's hands during the electron microscopy studies.

The films showed a few "mica particles" which were introduced during stripping from the cleaved mica surface. These particles, which can be readily recognized by their characteristic images and electron diffraction patterns, are practically the only contaminants found in the control specimens. The film surfaces were extremely smooth, uniformly clean, and devoid of background structure and particularly free of any recognizable (organic) contaminants. Electron diffraction patterns which were recorded from these films consistently feature the concentric diffuse ring patterns characteristic of carbon-silicon-monoxide-formvar substrates.

Electron micrographs of control thin films of formvar coated with carbon and silicon monoxide (Fig. C-2) were taken from a large series of prints, systematically recorded at higher electron optical magnifications to map out the entire visible specimen film area in each control sample. These film surfaces were also very smooth and consistently free from any recognizable particulate or droplet contaminants, especially of the (organic) type frequently seen in the exposed "Luster" specimens featuring a marked sensitivity to electron beam irradiation. Except for occasional "holes" visible in the underlying formvar substrate, these films are of exceptional uniformity and coherence. The holes were characterized by very clean margins which did not show the typical droplet patterns commonly found in certain of the exposed Luster specimens. The micrographs were recorded with low intensity microbeam illumination and special cold stage (liquid nitrogen, type Armbruster) under controlled conditions giving a reduced contamination rate. Exposures on 70mm Kodak high definition film or on Kodalith photographic film permitted uninterrupted recording of 70 to 90 frames without breaking the high vacuum, thus further reducing contamination problems.

Electron Microscopy and Diffraction Studies of Luster on-flight exposed Specimens:

A. Micron-size dense particles: Dense irregular particles found on thin films of the platinum specimen holders, exposed during the Luster Flight, measure about 10 x 15 microns and are characterized by ragged edges (Fig. L-1). The particles are completely opaque to electrons in the central region but reacted immediately to a beam of higher intensity. They seem to melt and shrink with obliteration of the sharp contours. A series of concentric sharp rings, resembling some of the patterns shown in Fig. M-2, were featured in selected-area electron diffraction patterns. However, only a few residual rings and intense spots could be recorded from the bombarded (and presumably recrystallized or modified) particles. Dense particles of this size are rare, since most dense particles encountered are in the sub-micron range. This type of particle has not been detected in any of the controls. Although the possibility that this particle is a contaminant appears doubtful, it can not yet be excluded. More extensive investigation is necessary before particles of this type can be definitely characterized and their probable origin established.

Electron micrographs were recorded (Fig. L-2) showing changes produced by irradiation of the dense irregular particles which are displayed in Fig. L-1. The characteristic changes noted in this group of micrographs occurred within a few seconds after sharply increasing the image current density of the illuminating electron microbeam.

The area adjacent to the particle, which already exhibited signs of cracking and puncturing of the film, promptly ruptured, and the dense particle began to "melt" or evaporate, during which a light "halo" formed around the particle. The jagged, angular contours of the dense particle are obliterated and it shrinks or becomes markedly distorted, finally tearing the underlying film. The core of the particle appears to be quite refractory to intense electron bombardment and the typical evaporation and recondensation of tiny droplets described by C. L. Hemenway and R. K. Soberman (Astronom. J. 67, 262, 1962) was not observed.

If more particles of this type can be found, it might be possible to try ultrathin sectioning with the Moran-type diamond knife and ultramicrotome (after appropriate embedding) in order to obtain better preserved samples for electron microscopy and electron diffraction analysis.

Spherical dense particles of the size shown in Fig. L-18 (ca. 10μ diameter) were seldom encountered. Under electron bombardment of moderate intensity part of the material evaporated and recondensed around the large particle, forming collections of minute spherical or polygonal particles (ca. 100 to 2,000Å diameter). Electron diffraction patterns recorded from these areas exhibit characteristic sharp ring patterns with spacings very similar to those obtained from Canyon Diablo meteorite particles and also to the diffraction patterns obtained from micrometeorite particles described by Hemenway and Soberman. They might correspond particularly to the Fe-Ni alloys (taenite and kamacite) and exhibit certain electron-optical phenomena (e.g., Lorentz deflections which are characteristic of ferromagnetic materials). In addition, characteristic small-angle patterns with spacings of 12-17Å were noted and are being further investigated.

B. Dense "Fluffy" Particles: Aggregated particles of a dense and "fluffy" nature were examined (Fig. L-3). This very irregular type is seen to consist at higher magnification of numerous dense and light particles, about 0.3 to 3 microns, associated with an amorphous diffuse material which seems to envelop the particles, forming a loose aggregate. The particles resemble, in some respects, the "fluffy" micrometeorites first described (Fig. 10, p. 259-260) by Hemenway and Soberman. The amorphous material is quite sensitive to intense electron bombardment and either evaporates away or transforms into frothy complexes (See Figs. L-13 to L-15). Some of the adjacent thin crystalline lamellae are undoubtedly mica contaminants, but the typical association with this amorphous material has not been found in the controls.

C. Submicroscopic Particles: Conglomerates of submicroscopic dense particles (ca. 0.02μ to 0.2μ) were examined (Fig. L-4 to L-8) and found to occur frequently in various states of aggregation at the edges of characteristic tears and fissures in the formvar-carbon films which are otherwise resistant. These unusual tears, which run transversely across the rigid platinum slits and are often interrupted by actual "holes" punched through the 400-500Å thick films, are in themselves noteworthy, since they were never found in the controls. These tears are unlike any other type of accidental film ruptures, and they are invariably associated with particles and amorphous, electron-sensitive material (note thickened edges coated with this component in Fig. L-4). Moreover, since each specimen grid was checked with the light microscope and the integrity of the film was recorded photographically, prior to transfer to the ultrahigh vacuum containers (Figs. 1-6), the typical ruptures occurring in only a limited number of particle-containing specimen supports must be regarded as significant evidence in support of extraterrestrial origin. The appearance of "flapped holes" of different sizes is also consistent with the assumption that we are dealing with low-velocity collisions, as was first suggested by Hemenway and Soberman (p. 260). Larger dense particles are less frequently found at the edges of holes.

Submicroscopic dense particles (about 0.5μ to 0.8μ diameter) were surrounded by a "halo" of amorphous material (Fig. L-5). This type of particle is also found singly, scattered over certain film areas, but usually near a cratered region or not far from other particulate aggregates. It is generally possible to distinguish between "electron dense" particles, and "light particles," but both share the close association with the amorphous material which, in many ways, resembles the organic lipid or hydro-carbon coating found regularly in electron microscopic studies of particles prepared from such systems.

Further analysis by selective extraction with organic solvents, or vacuum-distillation under controlled conditions, can not be adequately carried out on the formvar-carbon films of these specimen holders. However, this approach is quite feasible when dealing with specimens collected on freshly cleaved mica surfaces. Experiments along these lines are envisaged in attempts to obtain additional information on the nature of this amorphous component.

Electron micrographs were also taken of irregular dense particles shadowed with a thin layer of carbon to provide a protective coating against the effects of intense or moderate electron bombardment. This method also imparts a three-dimensional view of the particle configuration and displays some of the surface features which are usually obscured (Fig. L-6). This type of particle exhibits a fragmented layered structure. Since particles somewhat similar to these have been found in the "White Sands" control series, and they also resemble clay particles, it is tentatively assumed that at least some of these particles are terrestrial contaminants. However, further studies are required to differentiate between true contaminants and possible "silicate" extraterrestrial particles which might have been collected in the course of the Luster experiment.

The type of particle complex shown in Fig. L-7 is very similar to the minute particle systems found around the edges of film fissures and holes. A distinguishing feature of the complex, which was observed quite frequently, is the abundance of the amorphous material and extensive degree of dispersion of the embedded dense particles measuring only 200Å in the lower size range to about 5,000Å for the larger particulate components. The preponderance of the amorphous material sometimes obscures the boundaries of the individual constituent particles and gives the impression that we are dealing with much larger particles. Patches of this material are encountered in about one third of all specimen holders examined in our preliminary survey. However, these patches are particularly prominent in films with holes and fissures, indicating that the particle complexes might possibly be the "debris" or fragmented remnants of larger impinging particles. This particle complex can only be detected in the form shown in Fig. L-7 if special precautions are taken to reduce electron bombardment. Contamination effects must also be considerably reduced and the specimen protected during observation in the electron microscope by using liquid nitrogen specimen cooling devices.

The typical association of the dense amorphous material with the extremely small (minute particles only 100Å in diameter are predominant in many areas) dense particles is shown in Fig. L-8. Selected-area electron diffraction patterns showed a pattern of concentric sharp rings with discrete spot patterns ascribed to the (crystalline) particulate components. Precise measurements are now being carried out on these diffraction patterns and attempts will be made to determine the lattice parameters of the diffracting material. A characteristic small-angle pattern (reflections of ca. 8Å to 17Å) can also be recorded under appropriate conditions from these specimen areas.

Asymmetric particles with dense core and light coat are found singly or aggregated at the edges of tears and fissures in the formvar-carbon films (Fig. L-13). In addition, characteristic ruptures of the carbon film (ca. 400Å thick) are present which appear to correspond closely to the silhouette of ragged irregular particles. These ruptures, which were never found in the controls and occur in only a limited number of particle-containing "Luster" specimen supports, are consistent with the assumption that we are dealing with extraterrestrial particles impinging on the thin specimen films at relatively low velocity.

The asymmetric dense particles shown in Fig. L-14 often feature a rounded "nose-cone" configuration which may be related to their mode of impingement on the resistant thin specimen substrates. These particles of about 0.1μ to 0.5μ are frequently encountered in many of the "Luster" specimens. Particles of this type have not yet been detected in the control specimens.

D. <u>Submicroscopic "Droplets" and Electron Sensitive Amorphous Material:</u> Electron micrographs and electron diffraction patterns of dense submicroscopic droplets display characteristic patterns of minute (ca. 200Å to 0.3μ diameter) droplets (Fig. L-9) which are commonly found in direct association with the amorphous (organic?) material condensed in irregular patches along certain areas of the specimen films. The droplets are extremely sensitive to electron bombardment, and they can only be photographed in the form shown in Fig. L-9 by using microbeam illumination of low intensity and liquid-nitrogen, specimen anti-contamination devices. Many of the larger droplets in Fig. L-9 had already evaporated or collapsed into a pale foamy network under the beam. Droplets of this kind have hitherto not been found in any of the controls, but are instead frequently observed in the "Luster" specimens.

Although the probability of a spurious contaminant is being seriously considered, no significant supporting evidence for contamination has yet been found. Possible contamination sources include organic contaminants derived from the rocket system itself, or of other contaminants impinging on the exposed surfaces during the flight. A series of diffuse concentric rings and typical small-angle diffraction patterns were found in selected-area electron diffraction patterns.

Electron micrographs and electron diffraction patterns from areas of dense submicroscopic droplets showed the individual large droplets (Fig. L-10) to remain well preserved before the onset of the moderately higher electron image density which is normally required to clearly visualize the specimen on the fluorescent screen. A slight increase in electron bombardment suffices to completely modify the dense droplets which are either volatilized or blown up into a foamy aggregate.

Corresponding selected-area electron diffraction patterns were very difficult to record because the requisite focusing of the microbeam on the droplets almost immediately obliterates the pattern. Taking special precautions, however, a few useful small-angle patterns were obtained and are currently being evaluated.

Certain areas which contain the typical intact dense submicroscopic droplets were masked off so that diffraction patterns could be recorded from these sites only (Fig. L-11). Corresponding selected area high resolution (small-angle) patterns feature a number of reflections in the range of 7Å to about 18Å. However, these patterns are still not consistently reproducible and further improvements in recording techniques, with specimen cooling down to -180°, combined with image intensifiers of the Haine-Komoda type, will be required in order to obtain reliable data on this important component. When the peculiar amorphous material is found (either in droplet form or as a coating) in close association with particulate components (crystalline or amorphous) it becomes possible to satisfactorily record electron diffraction patterns using the underlying crystalline diffraction patterns as a useful guide in aligning the system for recording small-angle patterns. As shown in Fig. L-12, micrographs of the masked particles, surrounded by the amorphous material, and the corresponding selected-area, high resolution (small-angle) diffraction patterns are being recorded and evaluated from a number of representative areas. In addition to the single-crystal diffraction patterns (probably from mica flakes) there are a series of diffuse rings and reflections close to the central beam in the range of 7Å to about 18Å. Somewhat similar patterns were obtained from various cyclic (e.g., cholesterol) and eliphatic (e.g., paraffins, fatty acids, etc.) compounds.

Attempts are currently underway to identify the amorphous material by combined selective extraction experiments (e.g., with lipid solvents) and, particularly, by detailed comparison of these electron diffraction patterns with the patterns of the known aliphatic and aromatic hydro-carbons. These are being produced by Dr. E. Anders, R. Hayatsu, and M. H. Studier at the University of Chicago and Argonne National Laboratory, through aFischer-Tropsch type reaction between carbon monoxide and hydrogen using iron and stony meteorites as effective catalysts.

The type of particle shown in Fig. L-15 features a dense core surrounded by a coating of amorphous material which is very sensitive to the effects of electron-beam irradiation. There are also smaller particles which appear to consist entirely of this electron-sensitive material. After electron-beam bombardment they expand into characteristic "foamy" structures.

The characteristic electron-sensitive material shown in Fig. L-16 more closely resembles the "droplets" and amorphous material found frequently in the "Luster" specimens. When these areas were examined with micro-beam illumination of low intensity, using a liquid nitrogen cooling device, the dense droplets were relatively well preserved. However, upon slight increase of the electron-beam intensity, the droplets immediately volatilize and turn into typical "foamy" structures. This material, occurring most often at the edges of fenestrations in the carbon-formvar film, has not been detected in the corresponding control specimens.

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Collateral Model Experiments on Known Samples of Representative Iron and Stony Meteorites: As part of a comprehensive research program, these studies aim at applying and testing the same electron microscopic and diffraction techniques to known samples of representative iron and stony meteorites. Electron micrographs and electron diffraction patterns were taken of particles from the Canyon Diablo meteorite, kindly supplied by Dr. E. Anders. Examination of this material will provide a reference standard for parallel experiments designed to supplement the current electron-optical investigations of micrometeoroid and other extraterrestrial material collected by the rocket payload.

An important phase of this work is being carried out in close collaboration with Dr. Edward Anders, R. Hayatsu, of the Enrico Fermi Institute for Nuclear Studies, University of Chicago, and Dr. Martin Studier of the Argonne National Laboratory. These authors have recently found (P.I., 65-115, 1965) that iron and stony meteorites are effective catalysts for a Fischer-Tropsch type reaction between carbon monoxide and hydrogen. In hydrogen-rich mixtures approaching cosmic composition, aliphatic and aromatic hydrocarbons are produced rapidly at temperature between 25° and 580°C. It would be of interest to establish by electron microscopy and diffraction if there is any similarity with certain (presumably organic) components found in the collected micrometeoroid specimens. Recondensed material appeared near the particles following electron bombardment (Fig. M-1). Typical electron diffraction patterns recorded from these areas may correspond mainly to the Fe-Ni alloys (taenite and kamacite).

Electron micrographs and selected-area electron diffraction patterns were taken of particles from the Canyon Diablo meteorite, after acting as catalysts for a Fischer-Tropsch type reaction between carbon monoxide and hydrogen. In the collaborative experiments, ground Canyon Diablo meteorite powder was reacted with C-H-O mixtures in a quartz tube at 25°-580°C, yielding aliphatic and aromatic hydro-carbons. Minute quantities of the reacted meteorite powder were prepared according to the surface-film technique on a clean water surface,and the specimens were picked up on an ultrathin (50Å) carbon film for electron microscopy. In addition to areas containing metallic particles, numerous regions of the specimens feature light (crystalline) layers coated with amorphous material which volatilized under the electron beam. Selected-area diffraction patterns from such areas (Fig. M-2) exhibit patterns of sharp rings and typical small-angle patterns which are being further investigated.

Examined areas of this meteorite material, as pictured in Fig. M-3, featured conglomerates of fine filamentis metallic particles (whiskers?) associated with corrugated thin films. Selected-area diffraction patterns from such areas exhibit a characteristic pattern of sharp rings and small-angle patterns which are being further investigated. These patterns might possibly be correlated with oxidized material impregnated with relatively non-volatile hydro-carbons.

The same specimen shown in Fig. M-3 was examined at higher magnifications by high resolution selected-area diffraction pattern with central beam stop (Fig. M-4). These experiments illustrate the unique advantages of correlated electron microscopy and electron diffraction studies for direct visualization of the reaction products on submicroscopic micrometeorite particles under controlled conditions.

High resolution electron micrographs, along with selected-area electron diffraction patterns, were also taken of evaporated thin films of the Canyon Diablo meteorite as a control for the reacted specimens shown in Figs. M-6 to M-8. These experiments were also carried out in collaboration with Dr. Anders, R. Hayatsu, and Dr. M. Studier. The meteorite powder was first evaporated in an ultrahigh vacuum (10^{-8} mm Hg) on freshly cleaved mica, thus providing an ideal surface for subsequent electron microscopy studies. The thin film was then reacted with C-H-O mixtures in a quartz tube at 450° to 900°C., yielding characteristic aliphatic and aromatic hydrocarbons. The electron micrograph in Fig. M-5 shows typical particulate and polycrystalline structures yielding distinctive diffraction patterns which serve as a reference standard for the reacted material. Typical desposits (organic material) appeared on the polycrystalline thin film (Ni-Fe) in Fig. M-6. Characteristic high resolution selected-area electron diffraction patterns recorded from these reacted areas are being further investigated in an attempt to identify some of the characteristic aliphatic and aromatic hydro-carbons known to result from this reaction.

The characteristic high resolution dark field micrographs (Fig. M-8), recorded with oblique microbeam illumination, are expected to yield important additional information on the organic components of film.

F. <u>Exploratory experiments with "flash-shadowing" techniques to determine the feasibility of "in-flight" shadow casting of collected specimens:</u> These exploratory experiments are currently being carried out in attempts to improve existing techniques for the collection and identification of extraterrestiral matter. Electron micrographs were taken (Fig. E-1) of spherical latex particles and adjacent clay particles which were "flash-shadowed" with tungsten, using "exploding wire" techniques.

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If it were possible to coat incoming particles with a metal "shadowing" layer at the instant of exposing the collecting surfaces of the sampling instrument, then we would have actually obtained a reliable "submicroscopic snapshot" of the particle, including a three-dimensional molecular replica or "mold" for subsequent electron microscope examination. This procedure would not only label the particle as "extraterrestrial" but clearly distinguish it from any other contaminating material (which could have been previously marked by pre-flight shadowing with another metal).

In addition, labile components could presumably be encased and their surface replica faithfully preserved before they disintegrate or melt. Based on previous work with similar techniques, we are using very thin tungsten, iridium or palladium wire (1-4 mil Ø) placed close to the collecting surface of freshly cleaved mica. When a brief current pulse, triggered by a micro-switch (using simple miniature condenser discharge supply or small flashlight batteries), is passed through the wire, it will volatilize instantaneously, releasing enough metal vapor to give sharp shadows (even in a poor vacuum of a few microns). Resolution and stability of these deposits are superior to standard aluminum shadowing. The latex particles have a diameter of 268Å.

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DISCUSSION

In evaluating the results of this preliminary examination, the numerous possibilities of contaminating sources must be carefully considered as has already been stated by N. H. Farlow, M. B. Blanchard and G. V. Ferry of Ames Research Center in their comprehensive report. However, it should also be pointed out that the exemplary character of the precautions taken by the Ames group insures achievement of a cleanliness level which is considered to be unexcelled for this type of experiment.

It is against this background that our own experiments were designed and carried out under conditions of minimized specimen contamination and radiation damage which have hitherto not been attained in previous investigations. Thus, the use of platinum holders instead of the usual copper specimen grids, the collecting surfaces of carbon films prepared in an ultra-high vacuum, and the subsequent precautions taken during the actual electron microscopic examination, are all significant factors in evaluating the validity of these preliminary findings. In addition, the higher resolution inherent in our experimental design was actually realized in the course of an extensive examination of specimens (representing about 20% of the total areas available to us) carried out by two independent electron microscope groups. Moreover, the combined application of electron microscopy with high resolution electron diffraction of the same specimen areas extends the range and accuracy of our analytical techniques.

For example, in our experiment we were able to record electron micrographs permitting us to detect particles in the size range from 10Å-20Å up to 1000Å. In the preliminary electron microscope examination reported by N. H. Farlow, M. Blanchard, and G. D. Ferry ("Sampling the Leonid Meteor Stream with a Luster Sounding Rocket,"COSPAR paper) it is stated that "fifteen electron microscope grids from flight and control slides were examined at a magnification of 1,500 times to detect all particles larger than 0.1 micron." Under these conditions, therefore, it would hardly have been possible to detect clearly the large number of sub-microscopic (100Å-1000Å diameter) particles which are abundant in our own specimens. This significant difference in level of resolution and in minimized contamination conditions during the actual electron microscopic examination must be borne in mind when considering the statement that "every particle shape observed on flight-exposed surfaces, except one, was duplicated by particles on the nonflight controlled surface."

In our case, although we have observed in the duplicate controls (both White Sand and the Luster Controls) a large number of contaminating particles of unidentified origin, these were clearly different from the particles identified in the Luster flight exposed surfaces when studied at the higher resolutions available to us.

Nevertheless, the rigorous criteria established by the Ames Research group are considered to be a definite standard of reference for evaluation of all work in this field. Thus, while we are inclined to share the skepticism which exists concerning the extra-terrestrial origin of many of these particles, we wish to qualify the application of these criteria within the framework of the inherent methodological limitations (such as specimen substrate, electron optical resolution, and modifications during electron microscopic examination, etc.).

It is not generally realized that the combined application of high resolution electron microscopy and electron diffraction is particularly applicable to the study of the type of submicroscopic particles in the range of 100Å-1000Å which are most abundant in this type of specimen. For example, by means of selected area electron diffraction it should be possible to identify particles of this size in a precise and reliable fashion (i.e., comparison of electron diffraction patterns with known patterns, determination of crystallographic lattice parameters, etc.). Therefore, the electron optical examination in this case carries over into a range of spatial resolution and chemical identification which is not accessible at present to the microprobe and X-ray analytical techniques.

With these reservations in mind, and clearly aware of the indispensible need for further work, the results achieved so far are in general agreement with the results reported previously by Hemenway and Soberman (Astronomical Journal, 67, 256, 1962). As shown in the corresponding electron micrographs, there are definite indications of the presence of particles in the micron and particularly sub-micron size which are closely similar both as regards the electron microscopical appearance and the corresponding selected area diffraction patterns to those described by Hemenway and Soberman. There is a close similarity of the particles and the corresponding diffraction patterns with those observed from Canyon Diablo meteorites (e.g., compare Figs. L-18 with M-1). Although evaluation of these electron diffraction patterns is still being carried out, the preliminary results are very suggestive that we are dealing with iron-nickel alloys (taenite and kamacite). In addition, the presence of characteristic electron optical phenomena (e.g., Lorentz deflections and imaging of magnetic domains by appropriate electron optical arrangements) indicates that we are dealing with ferromagnetic components. These observations and collateral electron diffraction studies are useful in helping to differentiate these particles from other components having similar diffraction patterns. It is considered unlikely that particles having the characteristic shape and electron diffraction patterns of this type could result from contamination in the course of the experiment. The numerous characteristic "ruptures" (Fig. L-13) which were never found in the controls and which in fact cannot be duplicated by any known controlled experimental modifications, are consistent with the assumption that we are dealing with the effect of extra-terrestrial particles impinging on the thin specimen films and modifying them in a characteristic fashion.

The salient observation is the characteristic abundance of submicroscopic particles. Similar particles in the size range of a few hundred Angstroms had already been reported by Hemenway and Soberman. However, with our improved resolution and the use of the extremely smooth carbon substrate we have been able to consistently detect particles which are considerably smaller (100Å down to 20-40Å). This type of particle was only found in the on-flight surfaces, and was distinctly different from other types of random aggregates of sub-micron size particles which were observed in a few of the controls.

When dealing with particles of this size, measuring only 20Å to 100Å, we must bear in mind that we are actually visualizing clusters of only a few hundred atoms. If subsequent experiments confirm the existance of large numbers of these submicroscopic dense particles, presumably mainly of metallic origin, then these observations may be of significance in connection with the recent data on the "sporadic E" layers assumed to be composed entirely of metallic ions as reported by R. S. Narcisi, A. B. Bailey, and L. Della-Lucca of the Air Force Cambridge Research Laboratories, Bedford, Massachusetts, at the recent Seventh Annual Space Science Symposium in Vienna, Austria. Rocket sampling of two layers of the sporadic E regions showed them to be composed of the positive ions of iron, magnesium, calcium, and nickel, all metals found in meteorites. It is conceivable that the sampling surfaces exposed in our rocket experiment could have collected clusters of these ions which would presumably show up in the form of the 10Å-100Å dense submicroscopic particles.

The interesting and unexpected finding of an opaque electron sensitive material which appears to coat many of the dense submicroscopic particles is primarily a result of the application, for the first time in our experiments, of low-intensity micro-beam illumination and specimen cooling. Although these findings are very preliminary and subject to a number of uncertainties, it is interesting to speculate on the possibility that we may be dealing with certain characteristic organic compounds of the type described by Anders, Hayatsu, and Studier in their recent experiments. E. Anders, R. Hayatsu of the Fermi Institute for Nuclear Studies, University of Chicago, and M. Studier of the Argonne National Laboratories have recently found that iron and stony meterorites are effective catalysts for a Fisher-Tropsch-type reaction between carbon monoxide and hydrogen. In hydrogen-rich mixtures approaching cosmic composition, aliphatic and aromatic hydrocarbons are produced rapidly at temperatures between 25° and 580° C. In a series of preliminary experiments which we are carrying out in collaboration with these authors, we have been trying to establish by combined electron microscopy and diffraction if there is indeed a similarity between certain of the presumably organic components found in the collected micro-metereorite specimens and these organic compounds. Considering the small size of the micrometeroid particles and the ionizing conditions present in the fringes of the earth's atmosphere, this possibility is a very attractive working hypothesis for future experiments.

Another interesting possibility which must be considered in the interpretation of such labile components is the existence of (mixed) gas hydrates of organic compounds as first suggested by S. Miller (Nat'l. Academy of Sciences, 47, 1798, 1961). Miller specifically pointed out that hydrates of air may be formed in the high clouds of the earth either from metastable ice or super-cooled water. It is also relevant to recall in this connection that the sampling experiments of noctilucent cloud particles carried out by Hemenway, Soberman, Witt, et al, (Tellus, XVI, 84, 1964) indicated that "a significant fraction of the cloud particles appears to have been coated with terrestrial ice."

Perhaps the most significant conclusion that can be drawn at the present time from all of these preliminary observations is the manifest need for a considerable improvement and refinement of our methodology for collecting and identifying extra-terrestrial particles.

In particular, there is a pressing need for some reliable procedure of "in-flight shadow casting" of collected specimens which would permit us to examine labile specimens such as electron sensitive organic compounds or hydrates at the moment of impact on the surfaces, preserving their characteristic features upon re-entry. In this way, labile components could presumably be encased and their surface replicas faithfully be preserved before they disintegrate or melt.

Promising exploratory experiments (Fig. E-1) are currently in progress for in-flight shadow casting using an exploding wire technique. These experiments indicate that it is in principle possible to coat incoming particles with a metal shadowing layer at the instant of exposing the collecting surfaces of the sampling instrument. We could hereby actually obtain a reliable submicroscopic snapshot of the particle, including a three-dimensional molecular replica for subsequent electron microscopic examination. This procedure would be of particular importance in labelling the particle clearly as extra-terrestrial and thus distinguishing it from any other contaminating material. It is believed that these and related approaches will become essential for all future experiments of this type in view of the extraordinarily complex and manifold sources of contamination which make the interpretation of present results so uncertain.

Another area which deserves further exploration is the non-destructive study of the larger particles. This could be accomplished either by ultra-thin sectioning with a diamond knife and ultramicrotome, or perhaps even better by using the greater penetrating power of high voltage electron microscopes which permits the examination of metal specimens a few microns in thickness. A combination of high-voltage microscopy and electron diffraction with X-ray absorption microspectra microscopy (A. Engstroem, et al, and Lundberg, Exptl. Cell Research, 12, 198, 1956) are among the numerous promising approaches worthwhile considering for future work.

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B. <u>Continuation of Correlated Electron Microscopic and Electron Diffraction Studies of Certain Meteorites and of Pre-Cambrian Organized Systems</u>.

1. <u>Investigation of Orgueil Carbonaceous Chondrite</u>.

We have been arranging to continue electron microscopic and electron diffraction studies of certain meteorites (Orgueil carbonaceous chondrite) carried out with Dr. Edward Anders and Dr. Frank Fitch of the University of Chicago. Preliminary experiments indicate that the composition and structural relationships of its constituents can be determined by the high resolving power of the electron microscope. Various preparation techniques are being applied under carefully controlled conditions. These techniques include ultrathin sectioning with a diamond knife, mechanical and selective chemical dissociation, followed by density gradient separation, negative staining, shadow-casting, etc. The ultrastructural data will be correlated with parallel chemical studies of organic constituents and with the results of selected area electron diffraction analysis.

2. <u>Investigation of Pre-cambrian Rocks of Gunflint Chert Formation of Southern Ontario, Canada</u>.

We are also arranging to continue electron microscopical studies of Pre-cambrian organized systems. Preliminary investigations of nonferruginous cherts of the Gunflint formation of southern Ontario were carried out by electron microscopy in collaboration with Dr. Edward Anders and Dr, Fitch. Dr. S. A. Tyler of the University of Wisconsin and Dr. Barghoorn of Harvard University have reported (Science, Vol. <u>118</u>, P. 606, 1954) the occurrence of primitive lower plants in these Pre-cambrian rocks, which are the oldest (about two billion years) structurally preserved organisms that clearly exhibit cellular differentiation. Electron microscopy reveals the presence of filaments, tubular structures and membranes of apparent organic origin.

These studies are of great interest in the evolutionary scheme of primitive life, since they may furnish insight into the molecular organization of the oldest known preserved living systems, bearing also on the evolution of membrane ultrastructure.

Electron micrographs of these studies were reproduced in the 1965 McGraw-Hill Yearbook of Science and Technology under the section entitled, "Photographic Highlights", p. 86.

Preliminary experiments were successfully carried out for the first time with a simple electron microscope which can be used for transmission electron microscopy and electron diffraction, using high-field superconducting solenoid lenses in an open-air-core, liquid helium Dewar, preferably operating in the persistent current mode.

In a series of controlled, reproducible experiments, electron microscopic images have been recorded of test specimens with accelerating potentials of 4-8 kV, using a niobium-zirconium solenoid without pole pieces, operating at 32.2 kilogauss in the persistent current mode.

These first experiments have demonstrated over a period of 4-8 hours of continuous operation the exceptional stability of the images and their relatively high quality at magnifications of 50 to 100 X. This work was reported in a paper entitled "Electron Microscopy with High-Field Superconducting Solenoid Lenses", in Proceedings of the National Academy of Sciences, Vol 53, No. 2,445-451, Feb. 1965.

C. <u>Continuation of Experimental Program in Electron Microscopy Using High-field Superconducting Solenoid Lenses.</u>

The key to understanding the functional properties of subunit structure of membranes is the direct visualization of their fine structure at the highest attainable level of resolution with the electron microscope. Preparation artifacts and instrumental limitations have previously hindered reliable examination of membrane ultrastructure below the level of 20-30Å.

We have, therefore, continued systematic application of improved instrumentation and preparation techniques for the study of extraterrestrial matter and cell membranes by high resolution electron microscopy.

The main problems to overcome for attainment of the ultimate theoretical resolution of 2Å are the correction of lens aberrations, stabilization of lens excitation current, and accellerating of voltage.

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Further experiments were carried out with various types of electron microscopes using high-field superconducting solenoid lenses and accelerating voltages of 50,000 volts. This work was reported at the National Academy of Sciences meeting, April, 1965, and appeared as an abstract in Science, Vol. 148, No. 3670, entitled "Electron Microscopy: Application of High-Field Superconducting Solenoid Lenses". These experiments served to confirm the exceptional stability of the images and their high quality under carefully controlled conditions.

Other instrumental advances have been made with coherent microbeam illumination, using pointed filaments and improved electron gun design. These improvements have made it possible to observe directly the structural detail in uranium-stained specimens with a point resolution of 2.8Å, as compared with previous values of 4-6Å.

With these present advances in the generation of stable superconducting electro-magnetic fields and low-temperature electron microscopy, it is conceivable that the contemplated design of high-resolution cryo-electron microscopes immersed in a liquid helium cryostat using superconducting electro-magnetic lenses and related types of miniaturized electron microscope systems, may find direct application in the examination in situ of lunar and planetary matter by electron optical techniques. In view of the high vacuum and low-temperatures already available in outer space such a cryo-electron microscope could be miniaturized. By being of smaller size and invested with a far greater resolving power (200 to 1,000 X with useful magnifications of 10^5 to 10^8S) than a light microscope, as well as the fact that the image can be directly transmitted by appropriate vidicon television and telemetering techniques, it would permit a greater range of applications and thus supplement and extend the presently contemplated automatic light microscope system for use on a planet.

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Preliminary experiments have been made using superconducting lenses in combination with pointed filament sources which appear promising for recording holograms according to the Gabor diffraction microscopy and wave reconstruction principle (D. Gabor, Proc. Roy. Soc., London A 197, 454, 1949). Although there are still numerous experimental difficulties, the results obtained with these experiments and the observations on imaging phenomena with superconducting solenoid lenses are providing essential data for the design of new types of miniaturized cryo-electron microscopes immersed in a liquid helium cryostat and of a separate superconducting solenoid objective lens with appropriate pole pieces and stigmators which may be used in conjunction with modern high resolution electron microscopes to replace the objective lens.

We have proceeded with the development of a special liquid helium Cold Stage for experimental work with superconducting lenses of special design. This stage would fit into our present high-resolution microscopes (Hitachi 11-B and Siemens Elmiskop I & II) and would permit us to try out systematically different types of pole piece design.

We are particularly interested in experimenting with pole pieces of the rare-earth metals like Dysprosium, Holmium, and Erbium which become strongly ferromagnetic below 16°. Dysprosium has a higher saturation value (at about 40 kilogauss) at liquid helium temperatures than iron (saturation at about 20-25 kilogauss). By using suitably designed objective lens pole pieces of dysprosium instead of the standard iron pole pieces, it should thus be possible to obtain stronger objective lenses of much shorter focal length and generally improved characteristics, in addition to the much higher stability of super-conducting lenses when operating in the persistent current mode.

Dysprosium is now commercially available, and we have already established contact with Dr. John Hulm and Dr. Stanley Autler of the Cryogenics Systems Department of Westinghouse to try to obtain these materials. In fact, we anticipate a fruitful collaboration with this research group which has pioneered in the field of superconductivity and superconducting magnets, and this goal is gradually becoming feasible.

When the basic characteristics and the anticipated superior performance of superconducting objective lenses for high resolution electron microscopy have been clearly established through these indispensable exploratory investigations, we shall proceed to develop the other components of the miniaturized cryoelectron microscope.

Our work on the development of the cryo-electron microscope has resulted in a joint effort with the A. D. Little Company which promises to be fruitful. Based on our earlier work with Prof. Samuel Collins, former Professor Emeritus of MIT and now consultant with A. D. Little, we have been working on the design and operation of a closed circuit liquid helium cryostat. This machine, designed for continuous unattended operation, is ideal for supplying the cryogenic refrigeration required to cool a superconducting device.

If this closed cycle ADL CRYODYNE-(R) Helium Liquifier is successful, a highly profitable arrangement will thus be made available, not only to our project, but to electron microscopists and other groups who have need of a continuous liquid helium refrigeration without vibrations. The convenience of this device becomes obvious when one compares the present weekly cost for 100 liters of helium, around $700, with the estimated $30 per week in operating costs with the new CRYODYNE-(R).

D. Investigations of Macromolecular Organization of Hemocyanins and Apohemocyanins.

Investigation of the macromolecular organization of hemocyanins was carried out in collaboration with Dr. Ernst van Bruggen, an International Postdoctoral Fellow of the National Institutes of Health. High resolution electron microscopy was applied to these studies in combination with a variety of techniques. Included were low-temperature electron microscopy, negative staining, examination with coherent microbeam illumination, and high resolution shadowcasting.

Detailed elucidation of the macromolecular organization of the hemocyanins, the important oxygen-transporting blood proteins, at a level of resolution close to their quaternary structure, and the novel experimental approaches jointly undertaken with Dr. van Bruggen, are of direct relevance to our understanding of multienzyme complexes and membrane organization in general.

The joint studies are described in detail in the following papers which have been accepted for publication in the Journal of Molecular Biology, 16, p. 191, March, 1966.

1. H. Fernández-Morán, E.F.J. van Bruggen, M. Ohtsuki, Macromolecular Organization of Hemocyanins and Apohemocyanins as Revealed by Electron Microscopy.

Comparative electron microscopic studies of the structural organization of representative hemocyanins and apohemocyanins from Mollusca and Arthropoda are described. *Helix pomatia*, *Busycon canaliculatum*, and *Loligo pealei* were chosen as examples of the Mollusca; *Homarus americanus* and *Limulus polyphemus* represented the Arthropoda. Mollusca hemocyanins are cylindrical molecules (diameter about 340Å, height ranging from 140Å to 680Å) built up from 3 to 12 rows of subunits. Arthropoda hemocyanins are built from a cubic monomer (105Å) in various stages of organization which is species dependent. Mollusca hemocyanins are distinctly different from Arthropoda hemocyanins, although they seem to be built from analogous subunits. Observations indicating the possible presence of certain constituents specifically localized in the core of the Mollusca hemocyanins are discussed.

A differentiated outer layer is regularly found around the hemocyanin and apohemocyanin molecules of Mollusca. It is not known whether these structures are actual components of the native molecules or are determined by the preparation techniques. Their possible presence can have significant biological implications. Reproducible differences between hemocyanins and apohemocyanins were observed only in the Mollusca and under certain conditions. High resolution electron microscopy using improved preparation techniques and instrumentation (coherent microbeam illumination) revealed new structural details in the molecules close to quaternary levels. The results are discussed in relation to the available biochemical and biophysical data on these highly organized macromolecular complexes.

2. E. F. J. van Bruggen, H. Fernández-Morán, Reassociation of Hemocyanins from Subunit Mixtures.

   The dissociation and reassociation reactions of hemocyanin mixtures were studied by electron microscopy. The experiments were done respectively with a mixture of _Helix pomatia_ and _Loligo pealei_ hemocyanin (both belonging to phylum Mollusca) and with a mixture of _Helix pomatia_ (a Mollusc) and _Limulus polyphemus_ (an Arthropod) hemocyanins. After reassociation many of the original molecular structures are observed together with a certain amount of smaller and irregularly aggregated material. The importance of these specific reassociation reactions between hemocyanin subunits from different classes and from different phylums is discussed.

E. _A program was carried out with Dr. Robert Haselkorn and his associates in the Department of Biophysics on the investigation of cell-free protein synthetic capacity in extracts from bacteria, virus-infected bacteria, and animal cells._

1. The electron microscopic and biochemical characterization of Fraction I protein, carried out in collaboration with Dr. Haselkorn, is of particular importance, because this protein with enzymatic activity comprises by weight at least 50 percent of the soluble proteins of chloroplasts. It represents the first occasion upon which the substructure of a protein enzyme of only 120Å diameter has been resolved by electron microscopy.

This work is reported in the following paper which appeared in Science, Vol. 150, pp. 1598, December 1965: R. Haselkorn, H. Fernández-Morán, F. J. Kieras, and E.F.J. van Bruggen, Electron Microscopic and Biochemical Characterization of Fraction I Protein.

High resolution electron micrographs of Fraction I Protein from Chinese cabbage leaves have been obtained. The protein, which has ribulose 1, 5-diphosphate carboxylase activity, appears to be a cube with edge of about 120Å. Substructure can be seen in individual particles, consistent with a model having 24 subunits, the number prescribed by the available physical and chemical data. This is the first occasion upon which the substructure of a protein enzyme of only 120Å diameter has been resolved by electron microscopy.

2. In collaboration with A.J.E. Colvill and E.F.J. van Bruggen, the physical properties of a DNA Dependent RNA polymerase from *E. coli* were studied. High resolution electron micrographs of *E. coli* RNA polymerase, relatively free of nucleic acid, have been obtained. The 18S and 25S species of polymerase appear to be one and two hexagonal discs, respectively. A smaller, four-subunit square structure was also observed.

In collaboration with H. Manor, E.F.J. van Bruggen, and R. Haselkorn, investigations were made of methionine relaxed particles from *E. coli*. The ribonucleoprotein particles that accumulate in *Escherichia coli* K22 W6 when starved for methionine have been examined in the electron microscope. They appear to be heterogeneous, without well-defined substructure. Well-defined particles, probably RNA polymerase, are a major contaminant of the preparations.

F. Experimental Work with Pointed Filaments.

We have continued experiments on improved electron sources using pointed filaments of oriented single-crystal tungsten, tantalum, rhenium, and niobium tips in new types of molybdenum cathode caps with replaceable thin molybdenum foil aperture (0.5mm Ø). These point cathodes used with double-condenser system provide stable microbeam illumination of high specific brightness (5-10 x 10^5 A cm^{-2}. $sterad.^{-1}$), small spot size, low velocity spread, and high coherence. Optimum design parameters for microbeam illumination, which are now being implemented as part of our comprehensive research program in high resolution, low-temperature electron microscopy, include T-F emission in a molybdenum gun operating in ultrahigh vacuum.

This work was carried out in collaboration with Dr. Keiji Yada, a visiting professor from Tohoku University in Japan. After special work on tantalum pointed filaments, he succeeded in resolving the lattice spacing of (200) planes in NaCl crystals, measuring 2.815Å by direct axial illumination.

G. Special Biological Systems for Research Program in Low-Temperature Electron Microscopy.

1. Experimentation with Perognathus longimembris (pocket mice).

At the suggestion of Dr. George Jacobs and through the courtesy of Dr. R. Lindberg, Northrop Space Labs, Hawthorne, California, we have acquired pocket mice to begin experimentation in co-ordination with our low-temperature electron microscopic studies of the Nervous System.

2. Experimentation with Artemia (shrimp) eggs.

Also at the suggestion of Dr. George Jacobs and through the courtesy of Dr. H. Morowitz, Department of Molecular Biology and Biophysics, Yale University, we have acquired Artemia eggs which will be included in the low-temperature electron microscopic studies of the Nervous System.

H. Preparation of Atlas: "Elements of Biomolecular Organization."

I am preparing a monograph-atlas entitled "Elements of Biomolecular Organization" which describes salient features of the molecular organization of virus structures, multi-enzyme complexes, and particularly, cell membranes and their derivatives. The atlas is being published by Springer-Verlag in Heidelberg, Germany and will appear first in English, followed by German and Spanish editions. This atlas is the only one of its kind at present and it should elicit the interest of biologists, physicists, chemists. It is a welcome opportunity to acquaint the broader scientific community with the work in our laboratory.

II. Organization and Operation of the Special Electron Microscope Laboratories in the Department of Biophysics, The University of Chicago.

With funds provided by The University of Chicago, NASA Grant (NsG 441-63), NIH Grants (B-2460, NB-04267, GM-13243), and AEC Grants (AT 30-1-2278, AT 11-1 1344), a special laboratory facility for high resolution electron microscopy has been completed and put into operation in the Research Institutes. These laboratories occupy a total of about 4,000 square feet and comprise:

A. 2,500 square feet of remodeled space in the basement with installation of special floors, wall partitions, ceiling panels, air conditioning of the type used in "clean rooms" for modern electronic industrial facilities. These laboratories are equipped with three electron microscopes with attached electron diffraction units. The facilities include ultra-high vacuum (Varian) evaporation units, four ultramicrotomes, light microscopes, and complete preparation and photographic darkroom facilities. All of the critical equipment has been installed on individual vibration control mountings of special design. Corresponding precautions were taken in the installation of non-magnetic stainless steel vintilation ducts, incandescent lights, and electrical conduit to minimize electrical and magnetic perturbations.

B. Adjoining laboratories of 920 square feet located on the second floor of the Research Institutes have been remodeled:

1. Room 203B (230 sq. ft.)

   This room has been prepared for storage of specimens, equipment, and laboratory apparatus. A Harris Cascade Refrigeration Biological Storage Machine was installed which operates at temperatures as low as -120°C.

2. Room 205 (230 sq. ft.)

   This room has been prepared as a site for superconducting experiments. A Siemens Elmiskop EM-II with accessories was installed. Darkroom equipment has also been installed to expedite development of plates taken during experiments in this room.

3. Room 207 (460 sq. ft.)

An X-ray diffraction unit with Kratky Camera was installed. Two vacuum pumping units were developed and installed which pre-pump photographic plates (at a rate of 912 plates per 2 hours), also with capacity to pre-pump 70mm film and camera. The efficiency of these pumps is such that it reduces working time by several hours. Plates, film, and camera were previously pre-pumped in the microscope itself which involves a much longer time.

C. An additional laboratory (Room P-III) was constructed in the clean room laboratories of the basement to house a Hitachi Perkin Elmer electron microscope and accessories. This microscope was installed and includes a double condenser lens, electron diffraction chamber, hot and cold stages, and image intensification system.

D. A special highly regulated power supply is located in an air conditioned enclosure on the fifth floor of the Research Institutes. This 50-kilowatt motor generator set, specially designed and manufactured by Westinghouse Company, is equipped with a new solid-state regulator, giving better than 0.1% voltage stability and very low harmonic distortion.

E. A special vibration-free room is now being built for installation of the new "cryo-electron microscope," to be adapted from the Hitachi 11-B electron microscope purchased with NASA funds. The ten-ton floating foundation and other features of this facility should make it possible to exploit the unique stability of superconducting lenses operating in the persistent current mode for long-term exposures: of the order of minutes to hours, instead of the 5 to 15-second exposures presently possible.

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III. Training Program.

A. Training has been carried out collaterally with and in addition to the various research projects. In particular, a course in Cell Ultrastructure (offered during the Spring Quarter, March - June, Tuesdays and Thursdays, 2-4 PM) is conducted for students and faculty of this university as well as from other institutions. (SEE ATTACHED LECTURE SCHEDULE). A corresponding lab course is held from 4-6PM.

B, The laboratory has served as a center for short-term training sessions and information exchange to other societies and institutions. Some of the key visits and lectures of the past year are listed below:

1. Dr. Bruce Montgomery
National Magnet Laboratory
Massachusetts Institute of Technology
April 11, 1966

Dr. Montgomery's visit concerned a proposed collaboration between himself and our laboratory to further develop our superconducting lens and cryogenics program.

2. Biomedical Career Conference Group. This was a group of outstanding high school students who received a toun through our laboratories and an introduction to the field of electron microscopy on November 20, 1965.

3. Midwest Electron Microscopical Society. Members of this group toured our laboratory and discussed electron microscopic techniques on December 10, 1965.

4. Participant in Hooke Lecture Series, University of Texas. Lectured on: "Cell Membrane Ultrastructure," and "High Resolution Electron Microscopy of Biological Systems," October 28 to November 11, 1965.

5. Dr. C. Harrison, Jr.
Dr. R. Dickson
Dr. J. Hung
Mayo Clinic
Rochester, Minnesota

These doctors made a two-day visit to our laboratories to study our instrumentation and preparation techniques for examining cell ultrastructure on March 24-24, 1966.

6. Lectured at Galileo Society, University of Chicago, January 10, 1966, on "New Approaches in High-Resolution Electron Microscopy."

7. Lectured at Louisiana Society for Electron Microscopy, New Orleans, March 4, 1966, on "New Approaches in High-Resolution Electron Microscopy of Biological Specimens."

8. Lectured at Industrial Diamond Association, Boca Raton, Florida, March 7, 1966 on "Applications of the Diamond Knife in Earth and Space Research."

9. Lectured at Anatomy Department, University of Chicago, March 11, 1966, on "Organization of Cell Membranes."

10. Guest lectured at 75th Anniversary Physics Colloquium, University of Chicago, March 17, 1966, on "New Approaches to High-Resolution Electron Microscopy."

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IV. List of Publications.

1. H. Fernández-Morán, Electron Microscopy with High-Field Superconducting Solenoid Lenses, in Proceedings of the National Academy of Sciences, Vol. 53, No. 2, pp. 445-451, February, 1965.

2. H. Fernández-Morán, Application of High-Field Superconducting Solenoid Lenses in Electron Microscopy, Abstract in Science, Vol. 148, No. 3670, April 1965.

3. H. Fernández-Morán, Electron Microscopy with Superconducting Lenses, to be published in McGraw-Hill Yearbook of Science and Technology, p. 249, 1966.

4. H. Fernández-Morán, Forms of Water in Biologic Systems and the Organization of Membranes, in Annals of the New York Academy of Sciences, Vol. 125, Art. 2, p. 739, 1965.

5. H. Fernández-Morán and E.F.J. van Bruggen and M. Ohtsuki, Macromolecular Organization of Hemocyanins and Apo-hemocyanins as Revealed by Electron Microscopy, Journal of Molecular Biology, Vol. 16, p. 191, March 1966.

6. E.F.J. van Bruggen and H. Fernández-Morán, Reassociation of Hemocyanins from Subunit Mixtures, Journal of Molecular Biology, Vol. 16, p. 208, March 1966.

7. R. Haselkorn, H. Fernández-Morán, F.J. Kieras, and E.F.J. van Bruggen, Electron Microscopic and Biochemical Characterization of Fraction I Protein, Science, Vol. 150, 1598, December 1965.

8. A.J.E. Colvill, E.F.J. van Bruggen, and H. Fernández-Morán, Physical Properties of a DNA Dependent RNA polymerase from E. coli, Journal of Molecular Biology, Vol. 17, April 1966.

9. H. Fernández-Morán, "Analytical Systems for Biological Study of Mars: The role of electron microscopy and electron optical techniques in Exobiology." Paper presented at Exobiology Summer Study on the Biological Exploration of Mars, Stanford University, Berkeley, California, August, 1964. Announced in Journal of Scientific and Technical Aerospace Reports by the National Aeronautics and Space Administration. Reference # SC/NsG-441.

10. McGraw-Hill, Inc. "Three New Designs Improve Electron Microscopes," Scientific Research, January 1966.

V. Acknowledgements.

It is a pleasure to thank Dr. Raymond Zirkle, Chairman, Department of Biophysics; Dr. William Bloom, Distinguished Service Professor Emeritus, Department of Biophysics; Prof. F. Schmitt, Massachusetts Institute of Technology; Dr. W. H. Sweet, Massachusetts General Hospital; and Prof. S. C. Collins, Massachusetts Institute of Technology for their valuable suggestions and whole-hearted support of our program.

We are indebted to Dr. Stanley Bennett, Dr. Leon Jacobson, Mr. Melvin Tracht, Mr. Vernon Annamunthodo, Mr. Donald Sigal, Miss Irene Fagerstrom, and Mr. Clement Mokstad for administrative guidance.

We are particularly indebted to Mr. R. Szara and Prof. L. Meyer of the Low Temperature Laboratory; Mr. Akerhaugen, Mr. John Costa, and Mr. Helmut Krebs of the Central Development Shop; and to Mr. John Hanacek and Mr. G. Gibson of the Machine Shop for their most valuable suggestions and technical assistance.

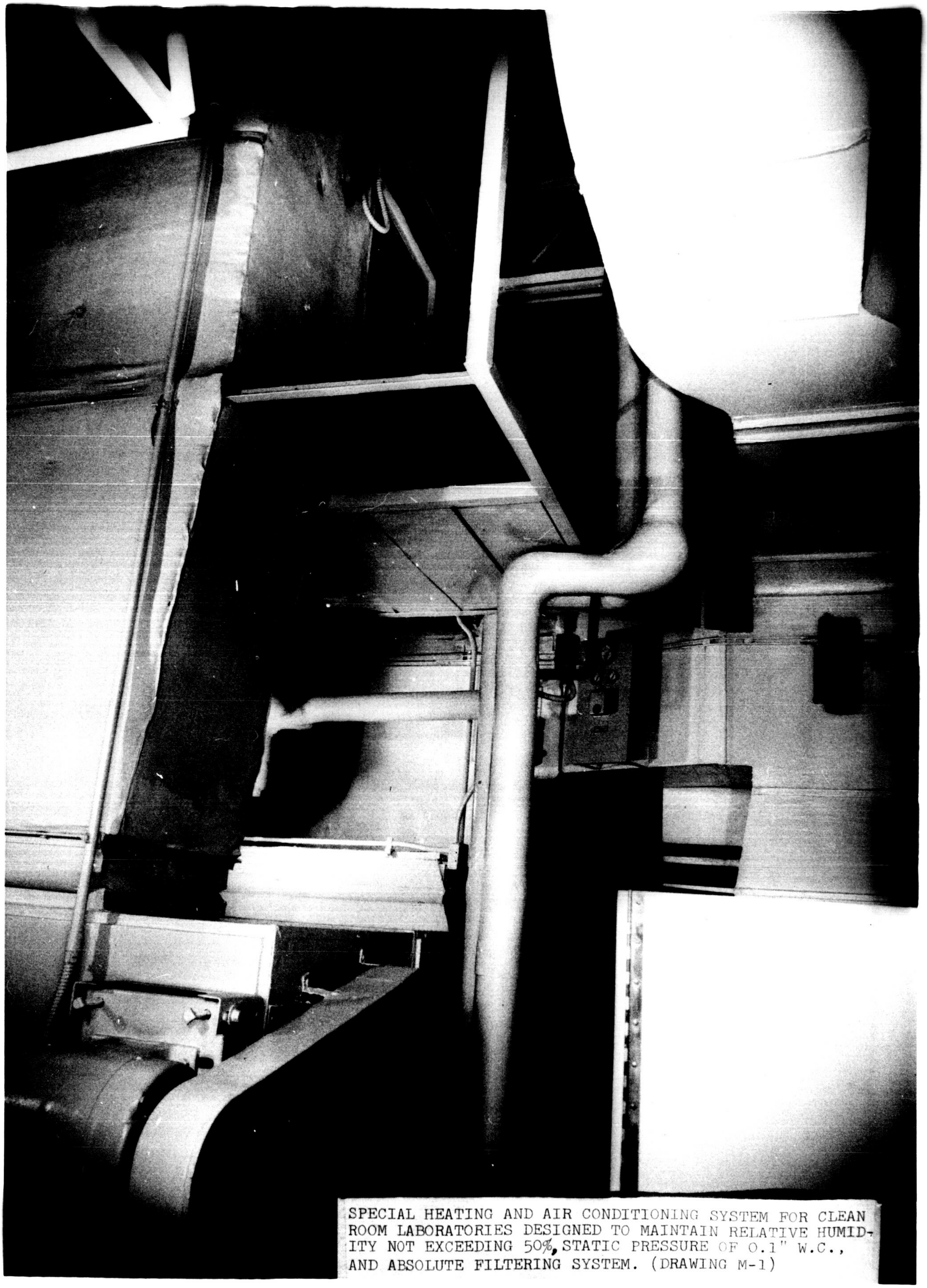
We wish to thank Mr. G. Olson and Mr. W. Connett of the Department of Buildings and Grounds for their kind help with our program.

We appreciate the valuable cooperation and supervision of Mr. C. L. Berrington, Mr. G. Monito, Mr. A. Peuron, Mr. D. Kasun, Dr. Stanley Autler, Dr. J. Hulm, and Mr. Fred Lins of the Cryogenics Systems Department of Westinghouse Electric Corporation.

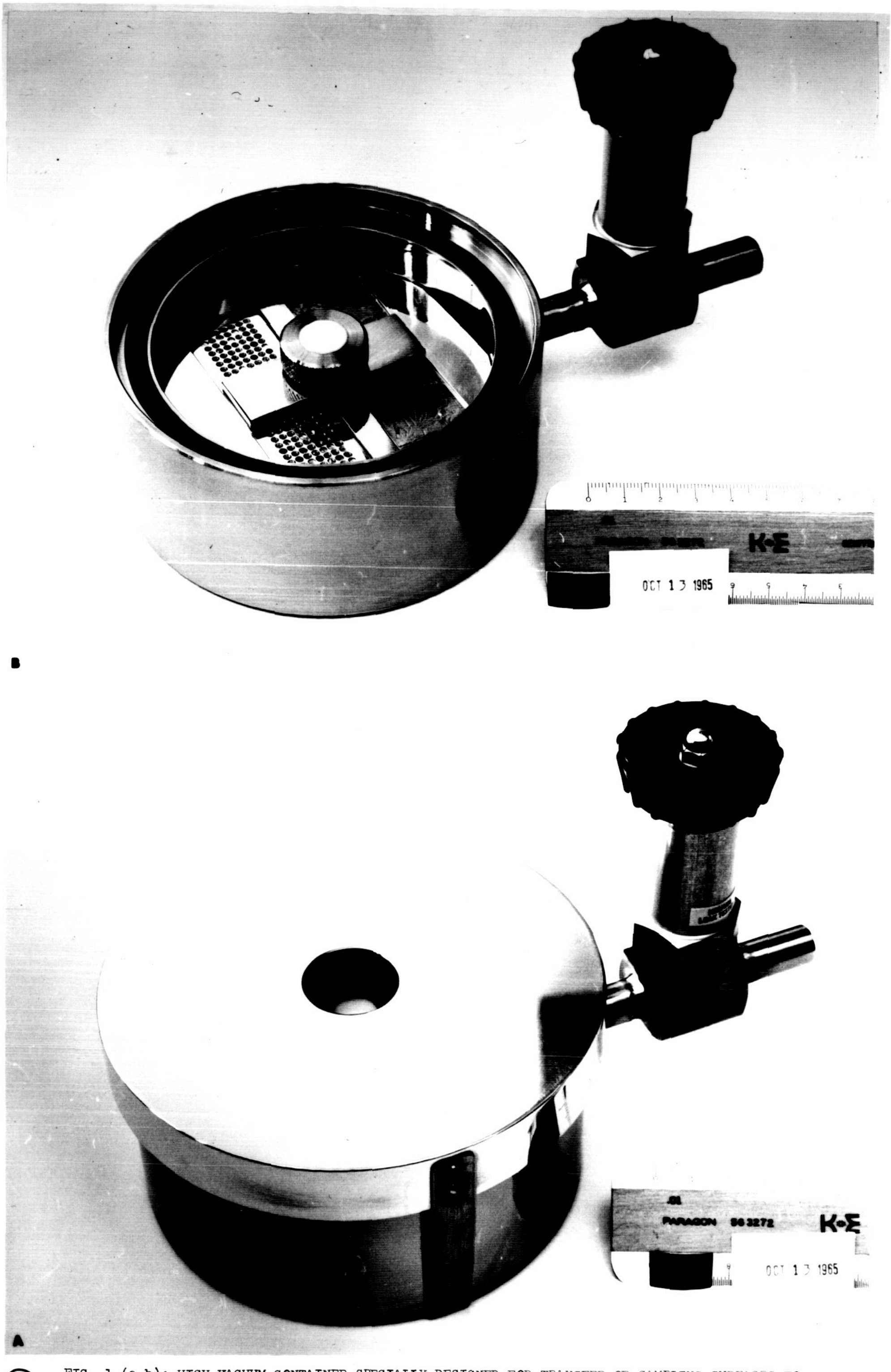
We are, of course, greatly obliged to our own staff: Mr. L. Ouwerkerk, Mr. R. Vicario, Mr. C. Hough, Mr. C. Weber, Mr. M. Ohtsuki, Miss D. Meddoff, Miss C. Runner, Mrs. Y. Shimada, Mrs. D. DeVere, and Mrs. S. Erikson. Special appreciation is due Mr. L. Sherman, graduate student, for his contributions to the program, and Miss J. Richardson for the compiling and editing of this progress report.

It is also a pleasure to thank Dr. George Jacobs, Dr. F. Quimby, and Dr. Orr Reynolds of the Bioscience Programs, National Aeronautics and Space Administration and Dr. Neil Farlow of the Ames Research Center, Moffett Field, California for their active concern and support for this project.

ENTRANCE TO ELECTRON MICROSCOPE LABORATORIES CLEAN ROOM AREA SHOWING SPECIAL WALL PARTITIONS, ENAMELED CEILING PANELS, RECESSED INCANDESCENT LIGHTS AND STAINLESS STEEL VENTILATION DUCT OPENINGS. ALL REASONABLE DESIGN FEATURES ARE INCORPORATED TO CARRY OUT HIGH RESOLUTION ELECTRON MICROSCOPY UNDER CLEAN ROOM CONDITIONS. (DRAWING A-1)



SPECIAL HEATING AND AIR CONDITIONING SYSTEM FOR CLEAN ROOM LABORATORIES DESIGNED TO MAINTAIN RELATIVE HUMIDITY NOT EXCEEDING 50%, STATIC PRESSURE OF 0.1" W.C., AND ABSOLUTE FILTERING SYSTEM. (DRAWING M-1)



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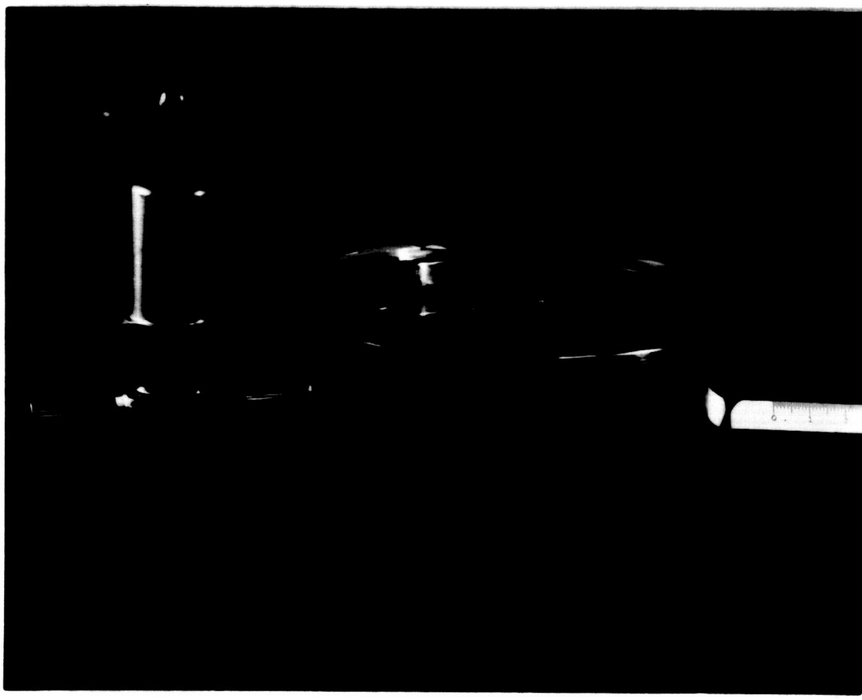
1 FIG. 1 (a,b): HIGH VACUUM CONTAINER SPECIALLY DESIGNED FOR TRANSFER OF SAMPLING SURFACES TO COLLECT EXTRATERRESTRIAL MATERIAL FOR ELECTRON MICROSCOPIC, ELECTRON DIFFRACTION AND MICROPROBE ANALYSIS UNDER CONTROLLED CONDITIONS OF MINIMUM CONTAMINATION. THIS STAINLESS STEEL CONTAINER PROVIDED WITH VITON GASKETS AND ULTRAHIGH VACUUM VALVE CAN BE LOADED WITH SEVERAL HUNDRED ELECTRON MICROSCOPE SPECIMEN HOLDERS, SINGLE-CRYSTAL MICA, PLASTIC AND OTHER THIN-FILM SURFACES UNDER RIGOROUSLY CLEAN CONDITIONS. IT IS MAINTAINED AT A HIGH VACUUM OF 10^{-7} to 10^{-8} mm Hg DURING TRANSPORT TO THE CLEAN-ROOM AREA WHERE ATTACHMENT TO THE MODULE PANS OF THE ROCKET-LAUNCHED PAYLOAD TAKES PLACE, AND THE SPECIMENS ARE SHIPPED BACK AFTER EXPOSURE UNDER SIMILAR HIGH-VACUUM CONDITIONS FOR DIRECT EXAMINATION BY ELECTRON MICROSCOPY. CONTAINERS AND SPECIMEN HOLDERS WERE DESIGNED, CONSTRUCTED, AND TESTED BY STAFF OF ELECTRON MICROSCOPE FACILITY, BIOPHYSICS DEPARTMENT, AND CENTRAL WORKSHOP OF THE RESEARCH INSTITUTES, UNIVERSITY OF CHICAGO. (Scale in mm.)



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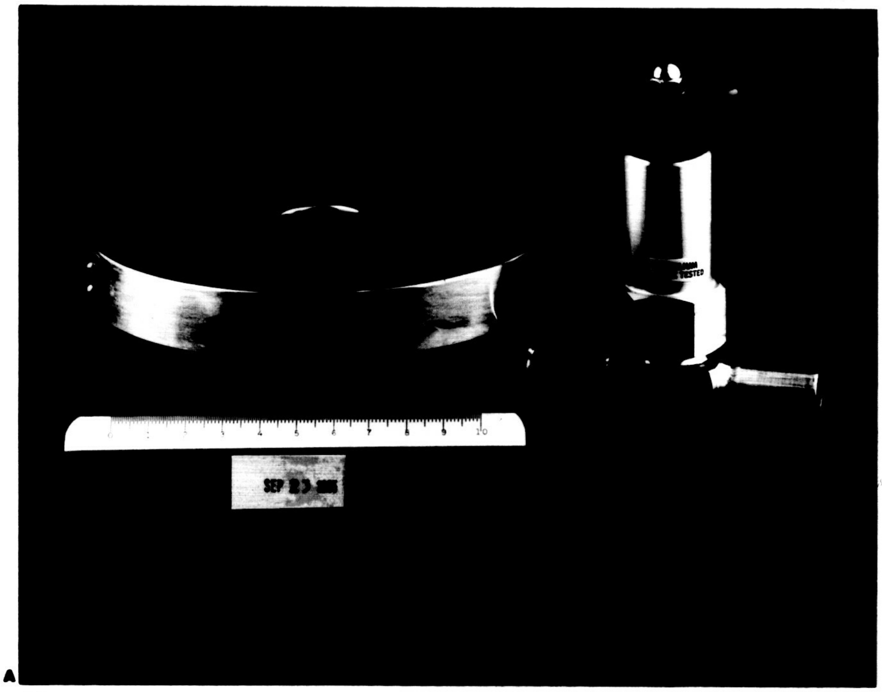

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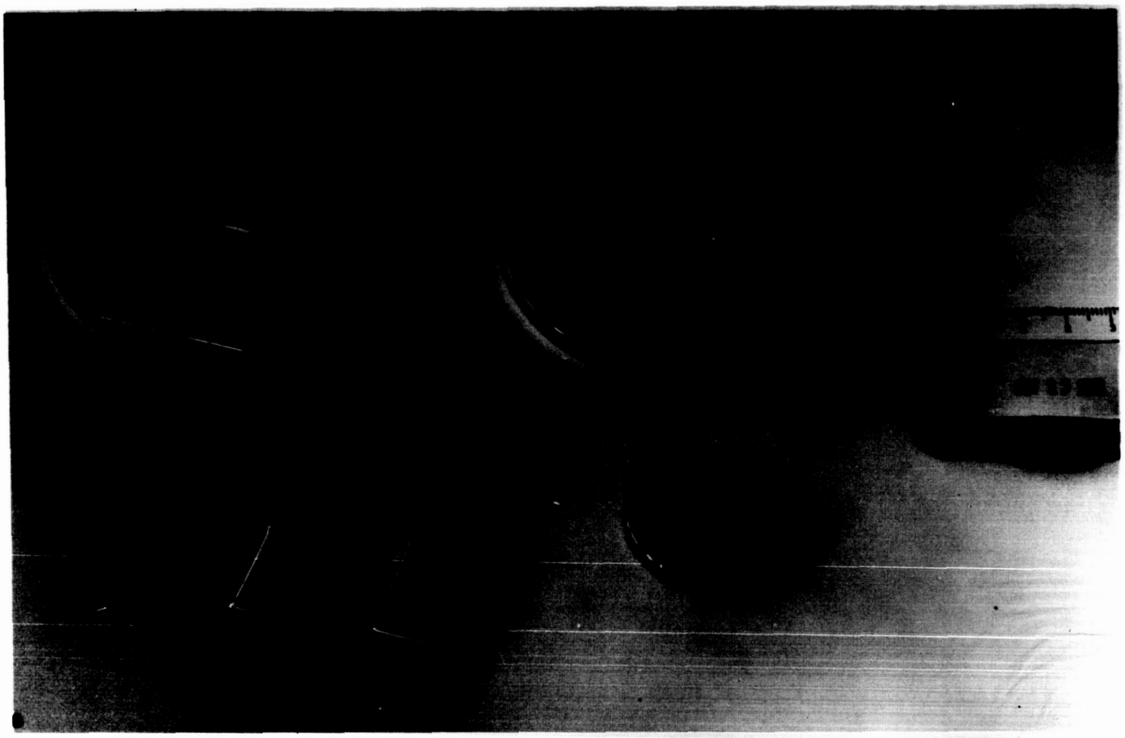
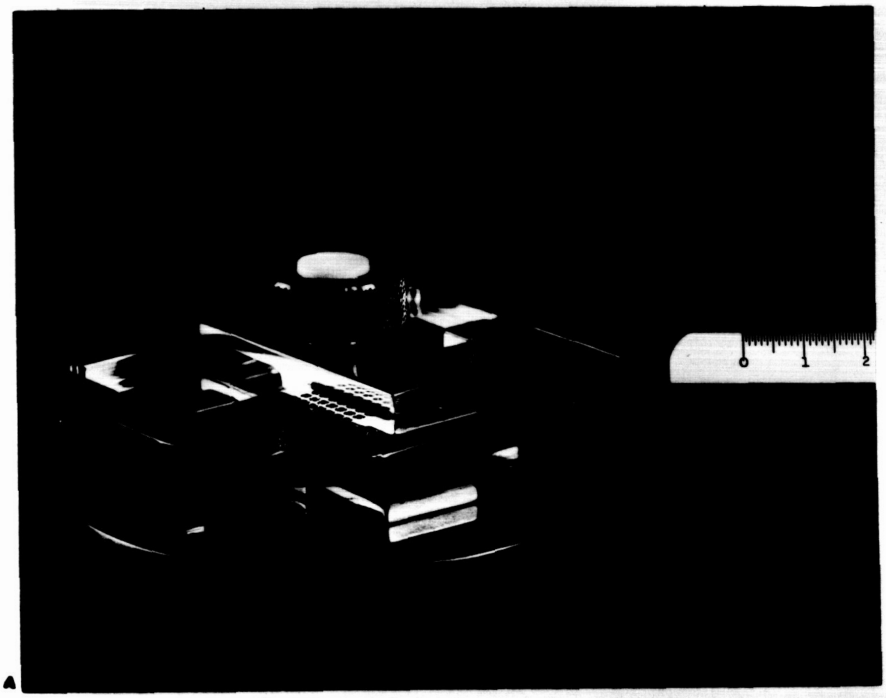
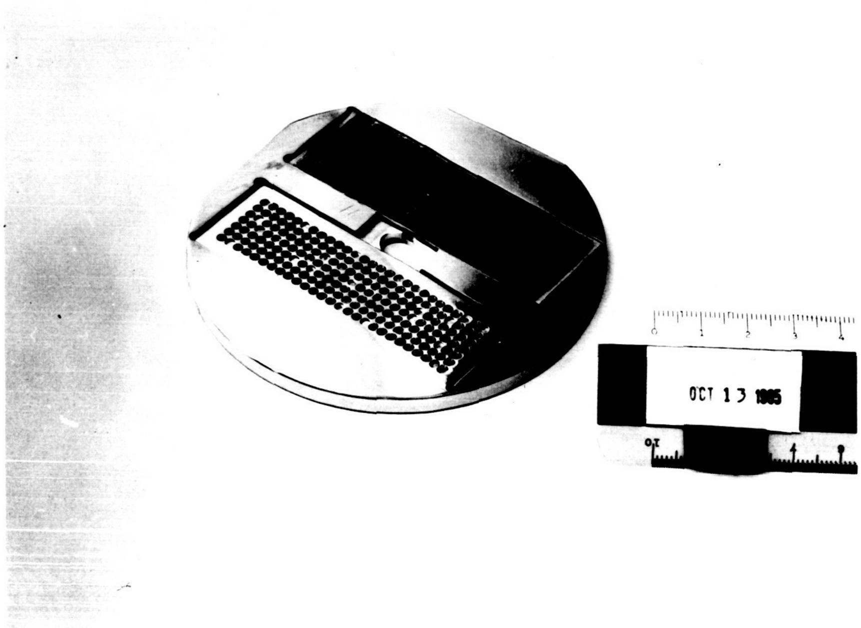
FIG. 2 (a,b): SPECIAL HIGH-VACUUM CONTAINER FOR TRANSFER OF SAMPLING SURFACES TO COLLECT EXTRATERRESTRIAL MATERIAL FOR ELECTRON MICROSCOPIC, ELECTRON DIFFRACTION AND MICROPROBE ANALYSIS UNDER CONTROLLED CONDITIONS OF MINIMUM CONTAMINATION. STAINLESS STEEL CONTAINER PROVIDED WITH VITON GASKETS, GLASS COVER PLATE, AND ULTRAHIGH VACUUM VALVE CAN BE LOADED WITH SEVERAL HUNDRED ELECTRON MICROSCOPE SPECIMEN HOLDERS OF PLATINUM AND OTHER TYPES COATED WITH THIN-FILM SURFACES OF CARBON, SINGLE-CRYSTAL MICA, AND APPROPRIATE ULTRA-CLEAN SURFACES OF SINGLE-CRYSTAL MICA FRESHLY CLEAVED, AND MAINTAINED THROUGHOUT AT HIGH VACUUM (10^{-7} to 10^{-8} mm Hg) FOR TRANSPORT TO CLEAN-ROOM AREA, ATTACHMENT TO MODULE PANS OF ROCKET-LAUNCHED PAYLOAD, AND SHIPMENT BACK TO ELECTRON MICROSCOPE LABORATORY. THREE IDENTICAL CONTAINERS WERE PREPARED FOR "LUSTER" PROJECT (Nov. 16, 1965) TO PROVIDE INDEPENDENT CONTAMINATION CONTROLS AT DIFFERENT SITES OF EXPERIMENTS TO BE CARRIED OUT AT ELECTRON MICROSCOPY LABS., BIOPHYSICS DEPT., UNIV. CHICAGO.





(3) FIG. 3 (a,b): HOLDERS IN SPECIAL HIGH-VACUUM CONTAINER FOR TRANSFER OF SAMPLING SURFACES TO COLLECT EXTRATERRESTRIAL MATERIAL FOR ELECTRON MICROSCOPIC, ELECTRON DIFFRACTION AND MICROPROBE ANALYSIS UNDER CONTROLLED CONDITIONS OF MINIMUM CONTAMINATION. THIS SIMPLE MODULAR DEVICE CAN BE READILY ASSEMBLED AND DISMANTLED WITH FEW MANIPULATIONS UNDER CLEAN-ROOM AND EVEN HIGH-VACUUM CONDITIONS. IT PROVIDES SECURE ATTACHMENT WITH SLIDES CONTAINING SEVERAL HUNDRED PLATINUM SPECIMEN HOLDERS COATED WITH THIN (100-300 Å) CARBON, SINGLE-CRYSTAL GRAPHITE OR MICA LAMELLAE, FORMVAR, SILICON MONOXIDE AND OTHER TYPES OF SUBSTRATES, IN ADDITION TO LARGER SURFACES OF FRESHLY-CLEAVED SINGLE-CRYSTAL MICA, PLASTIC SLIDES AND RELATED COLLECTING SURFACES SUITABLE FOR ELECTRON OPTICAL STUDIES. THREE IDENTICAL CONTAINERS AND SPECIMEN SUBSTRATE ASSEMBLIES WERE PREPARED FOR THE "LUSTER" PROJECT MICROMETEOROID SAMPLING FLIGHT SCHEDULED FOR NOVEMBER 1965 TO PROVIDE INDEPENDENT CONTAMINATION CONTROLS AT THE DIFFERENT SITES OF THE EXPERIMENT; AMES RESEARCH CENTER, ROCKET-LAUNCHING SITE, AND ACTUAL PAYLOAD SPECIMENS TO BE EXAMINED IMMEDIATELY AFTER RECOVERY AT THE ELECTRON MICROSCOPE CLEAN-ROOM LABORATORIES, DEPARTMENT OF BIOPHYSICS, UNIVERSITY OF CHICAGO.



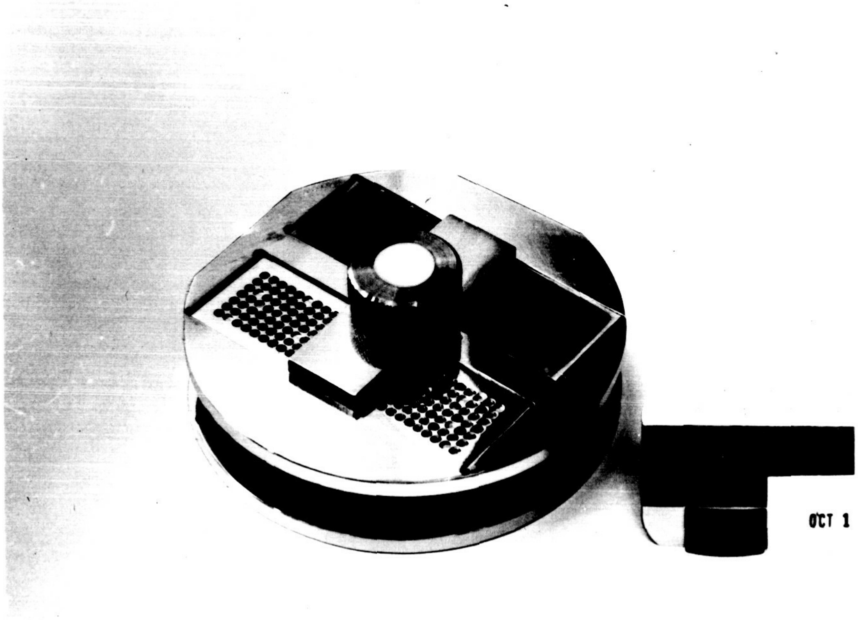


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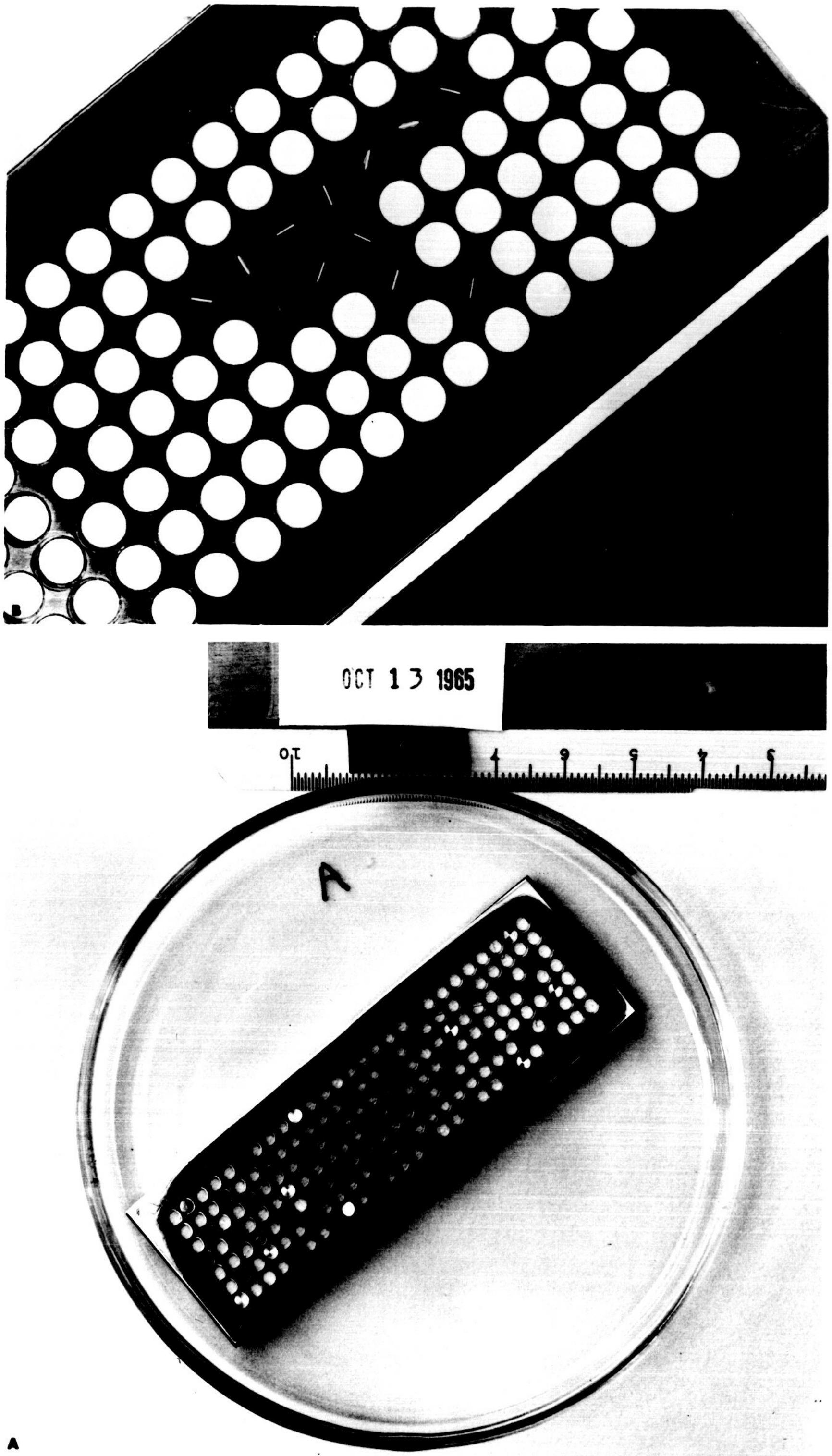
FIG. 4 (a,b): HOLDERS IN SPECIAL HIGH-VACUUM CONTAINER FOR TRANSFER OF SAMPLING SURFACES TO COLLECT EXTRATERRESTRIAL MATERIAL FOR ELECTRON MICROSCOPIC, ELECTRON DIFFRACTION AND MICROPROBE ANALYSIS UNDER CONTROLLED CONDITIONS OF MINIMUM CONTAMINATION. THIS SIMPLE MODULAR DEVICE CAN BE READILY ASSEMBLED AND DISMANTLED UNDER CLEAN-ROOM AND EVEN HIGH-VACUUM CONDITIONS FOR DIRECT TRANSFER OF SPECIMENS TO ELECTRON MICROSCOPE. IT PROVIDES SECURE ATTACHMENT FOR SLIDES CONTAINING SEVERAL HUNDRED PLATINUM SPECIMEN HOLDERS COATED WITH THIN FILM SUBSTRATES OF DIFFERENT TYPES, IN ADDITION TO LARGER SAMPLING SURFACES OF FRESHLY-CLEAVED SINGLE-CRYSTAL MICA, PLASTIC SLIDES AND RELATED COLLECTING SURFACES SUITABLE FOR ELECTRON-OPTICAL STUDIES TO BE CARRIED OUT AT THE ELECTRON MICROSCOPE CLEAN-ROOM LABORATORIES, DEPARTMENT OF BIOPHYSICS, UNIVERSITY OF CHICAGO. (Scale in mm.)



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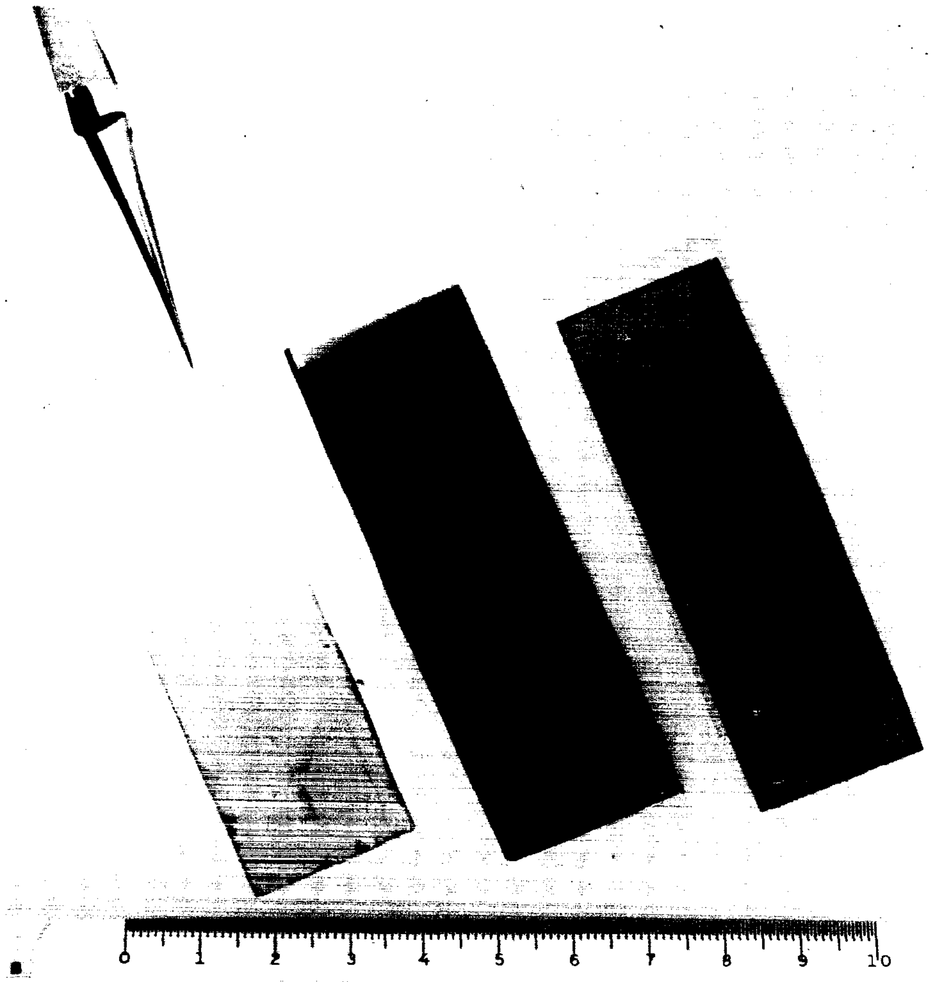
5 FIG. 5 (a,b): SLIDE WITH PLATINUM SPECIMEN HOLDERS IN SPECIAL HIGH-VACUUM TRANSFER CONTAINER TO COLLECT EXTRATERRESTRIAL MATERIAL FOR ELECTRON MICROSCOPIC,ELECTRON DIFFRACTION AND RELATED ELECTRON OPTICAL STUDIES UNDER CONTROLLED CONDITIONS OF MINIMUM CONTAMINATION.THE PLATINUM SPECIMEN HOLDERS WITH SLITS AND HOLES OF DIFFERENT CONFIGURATION ARE MORE SUITABLE THAN THE STANDARD THIN COPPER MESH ELECTRON MICROSCOPE SPECIMEN HOLDERS FOR THESE CRITICAL STUDIES. THE PLATINUM HOLDERS ARE COATED WITH ULTRATHIN (100- 300 Å) CARBON,FORMVAR,SILICON MONOXIDE FILMS,SINGLE-CRYSTAL GRAPHITE OR MICA LAMELLAE AND OTHER TYPES OF SUBSTRATES SUITABLE FOR HIGH RESOLUTION ELECTRON MICROSCOPY AND ELECTRON DIFFRACTION.THE FILMS WERE DEPOSITED IN A HYDRO-CARBON FREE,ULTRAHIGH VACUUM UNIT WITH VARIAN ION-PUMP (10^{-7} to 10^{-8}mm Hg.) IDENTICAL SPECIMENS WERE PREPARED FOR THE THREE CONTROL CONTAINERS AT THE DIFFERENT SITES OF THE EXPERIMENTS RELATED WITH THE "LUSTER" PROJECT SAMPLING FLIGHT SCHEDULED FOR NOVEMBER 1965.



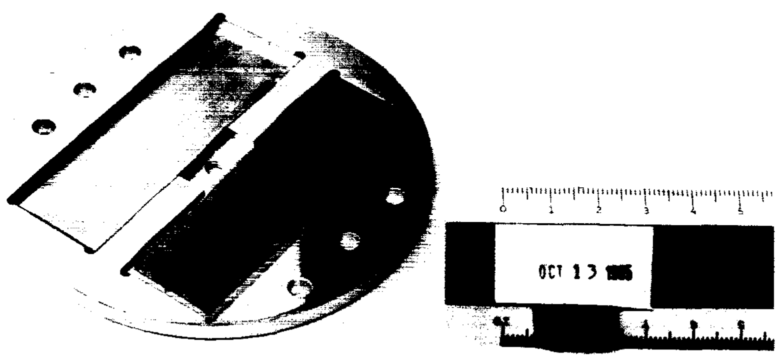


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FIG. 6 (a,b): SLIDES OF FRESHLY CLEAVED SINGLE-CRYSTAL MICA AND OF LUCITE IN SPECIAL HIGH-VACUUM TRANSFER CONTAINER TO COLLECT EXTRATERRESTRIAL MATERIAL FOR ELECTRON MICROSCOPIC, ELECTRON DIFFRACTION AND RELATED ELECTRON OPTICAL STUDIES UNDER CONTROLLED CONDITIONS OF MINIMUM CONTAMINATION. THE FRESHLY-CLEAVED SINGLE-CRYSTAL MICA SURFACES PROVIDE IDEAL, CLEAN AND ATOMICALLY SMOOTH SURFACES FOR COLLECTION AND EXAMINATION OF MICROMETEORITES AND EXTRATERRESTRIAL SPECIMENS. THE LUCITE SLIDE WOULD PROVIDE SUITABLE SUBSTRATES FOR EMBEDDING OF THE IMPACTING MICROMETEORITES WHICH COULD THEN BE SUBSEQUENTLY ULTRATHIN SECTIONED WITH A DIAMOND KNIFE AND ULTRAMICROTOME FOR ELECTRON MICROSCOPY AND ELECTRON DIFFRACTION (Fernandez-Moran, 1958-65). IDENTICAL SPECIMENS WERE PREPARED FOR THE THREE CONTROL CONTAINERS AT THE DIFFERENT SITES OF THE EXPERIMENTS RELATED TO THE "LUSTER" PROJECT SAMPLING FLIGHT SCHEDULED FOR NOVEMBER 1965. (Scale in mm.)

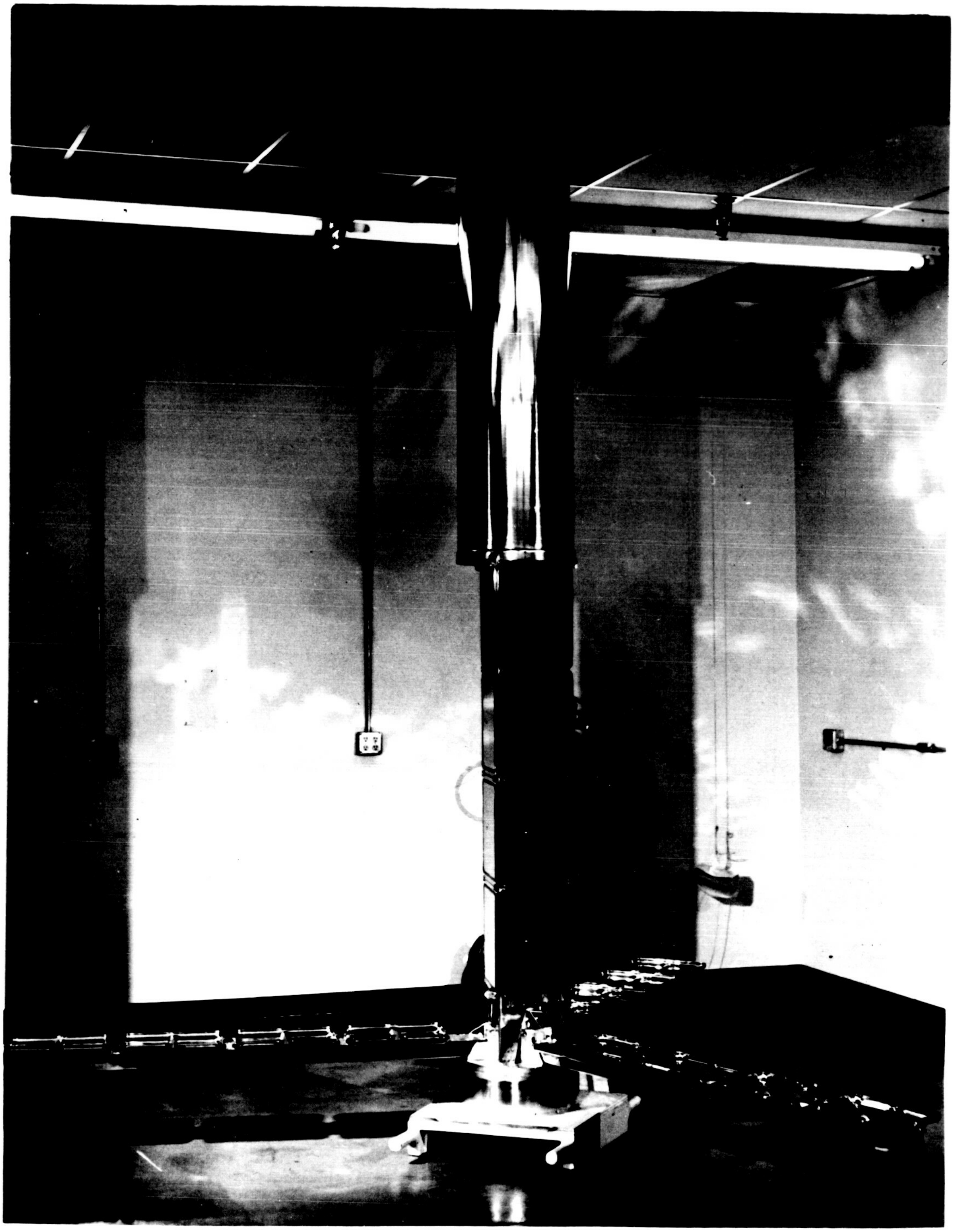


Figure 1 - LUSTER sampling instrument fully open.

Figure 2 - LUSTER module pans and covers attached to the sampling instrument

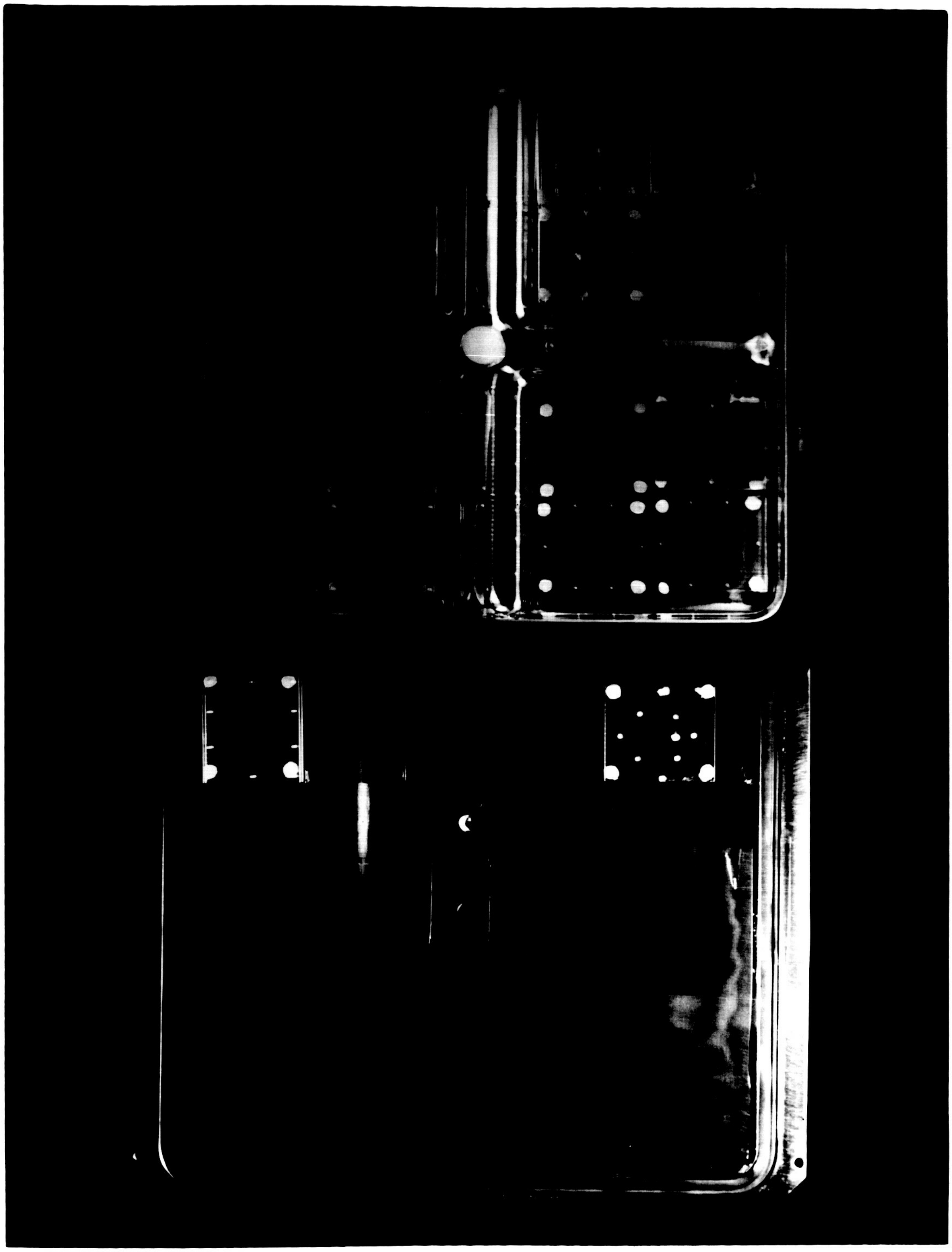
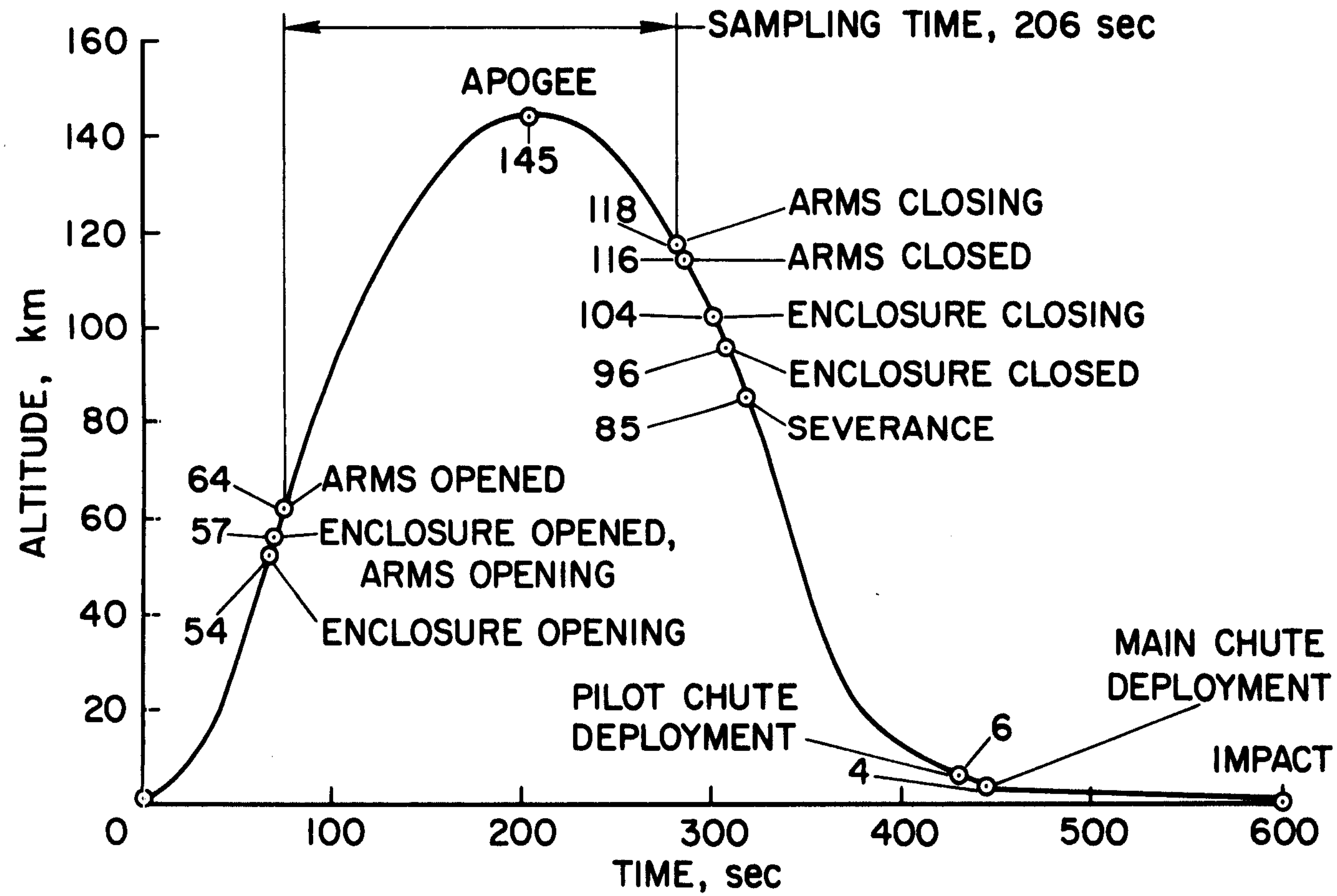


Figure 3 - Sampling surfaces attached to an open module pan and cover.

LUSTER FLIGHT, NOVEMBER 1965



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RECENT LEONID METEOR OBSERVATIONS

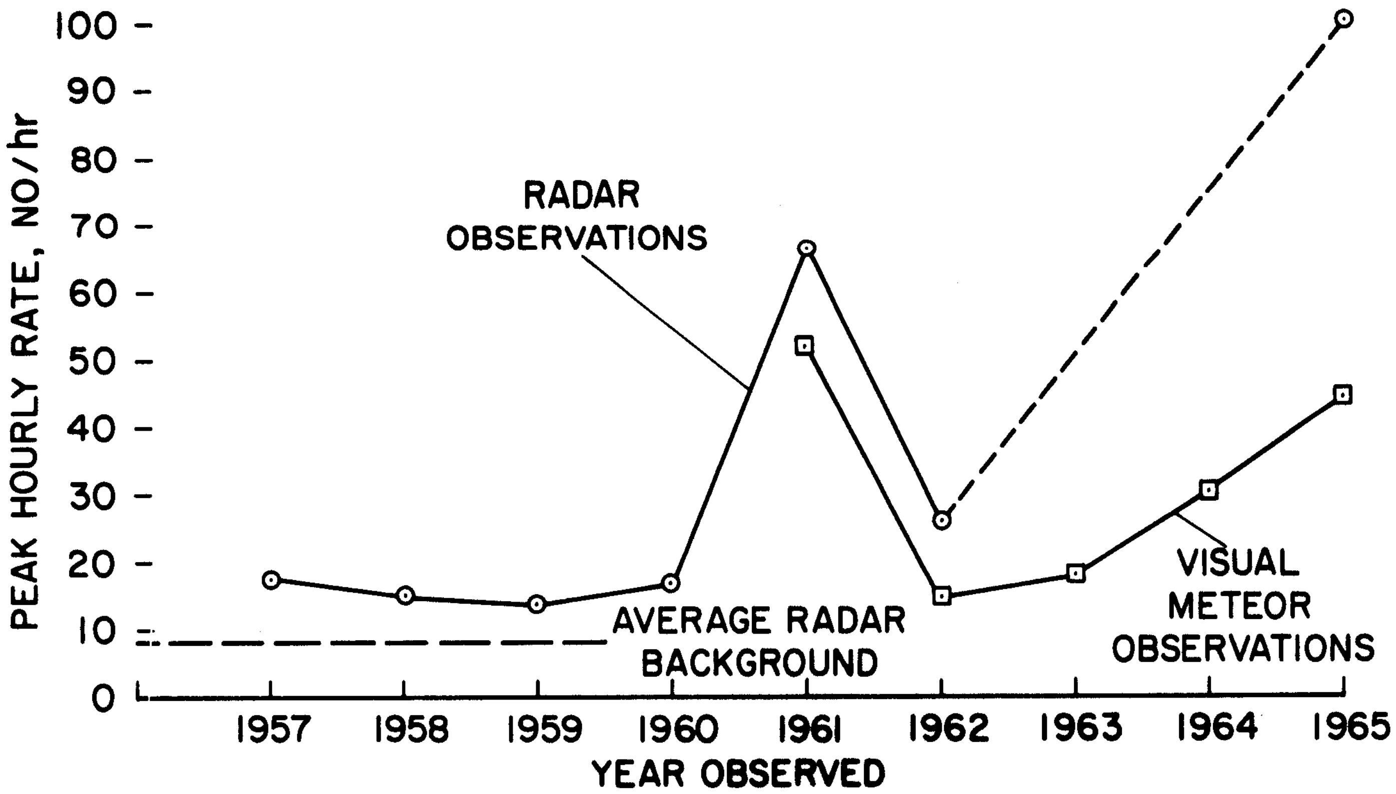


FIGURE C-1: ELECTRON MICROGRAPHS AND DIFFRACTION PATTERN OF REPRESENTATIVE CONTROL THIN FILMS OF CARBON-SILICON-MONOXIDE-COATED FORMVAR (ca. 200-400 Å) COVERING SLIT APERTURE OF PLATINUM SPECIMEN HOLDERS. ("Ames" Group Control Series No. E-23 & B-26). THESE EXCEPTIONALLY STABLE AND CHEMICALLY INERT SPECIMEN SUPPORTS ARE SUPERIOR TO THE STANDARD COPPER SPECIMEN GRIDS. THEY PROVIDE AN UNOBSTRUCTED VIEW OF THE ENTIRE SLIT AREA (ca. 1 x 0.1mm) WHICH IS FIRST SCANNED AND PHOTOGRAPHED AT LOW MAGNIFICATIONS (top view) UNDER CONDITIONS OF MINIMIZED SPECIMEN CONTAMINATION AND RADIATION DAMAGE BY USING ELECTRON MICROBEAM ILLUMINATION OF VERY LOW INTENSITY (image current density of about 10^{-11} to 10^{-12} amps/cm^2), AND A LIQUID NITROGEN SPECIMEN COLD STAGE. THE FILM SUBSTRATES WERE PREPARED BY EVAPORATION OF PURE CARBON AND/OR C-SiO IN ULTRAHIGH VACUUM (10^{-8} mm Hg) OF VARIAN UNIT WITH ION PUMP AND SORPTION FOREPUMP WITH LIQUID NITROGEN COLD TRAP, THUS ELIMINATING ANY POSSIBILITY OF CONTAMINATION WITH OIL VAPORS. THE FILMS ARE MOUNTED ON PLATINUM HOLDERS (FINAL CLEANING BY HEATING IN HIGH VACUUM) USING QUARTZ-DISTILLED, ULTRAFILTERED WATER AND REAGENTS OF CONTROLLED PURITY, AND OBSERVING THROUGHOUT RIGOROUS CLEANLINESS PRECAUTIONS IN SPECIAL CLEAN-ROOM ELECTRON MICROSCOPE LABORATORIES EQUIPPED WITH AIR CONDITIONING SYSTEM CONTAINING (CAMBRIDGE) "ABSOLUTE" FILTERS WHICH ARE EXPRESSLY DESIGNED TO PREVENT CONTAMINATION. EACH SPECIMEN HOLDER IS KEPT IN INDIVIDUAL SEALED MICRO-DESICCATORS AND PROVIDED WITH SEPARATE HANDLING DEVICES TO ELIMINATE ANY CONTACT WITH THE OPERATOR'S HANDS DURING THE ELECTRON MICROSCOPY STUDIES. THE CONTROL FILM DISPLAYED ABOVE SHOWS A FEW "MICA PARTICLES", INTRODUCED DURING STRIPPING FROM CLEAVED MICA SURFACE. THESE PARTICLES-WHICH CAN BE READILY RECOGNIZED BY THEIR CHARACTERISTIC IMAGES AND ELECTRON DIFFRACTION PATTERNS- ARE PRACTICALLY THE ONLY CONTAMINANTS FOUND IN THE CONTROL SPECIMENS. AS SHOWN (lower left) THE FILM SURFACES ARE EXTREMELY SMOOTH, UNIFORMLY CLEAN, DEVOID OF BACKGROUND STRUCTURE AND PARTICULARLY FREE OF ANY RECOGNIZABLE (ORGANIC) CONTAMINANTS. ELECTRON DIFFRACTION PATTERNS (lower right) RECORDED FROM THESE FILMS CONSISTENTLY FEATURE THE CONCENTRIC DIFFUSE RING PATTERNS CHARACTERISTIC OF CARBON-SILICON-MONOXIDE-FORMVAR SUBSTRATES. MICROGRAPHS AND DIFFRACTION PATTERNS RECORDED AT 80 kV, WITH POINT CATHODE OF SINGLE-CRYSTAL TUNGSTEN IN SIEMENS ELMISKOP IA HAVING SEPARATE OBJECTIVE LENS POWER SUPPLY OF IMPROVED STABILITY. MAGNIFICATIONS: (allowing for reduction in reproductions) Top view: 100x; Lower left: 12,000 x.

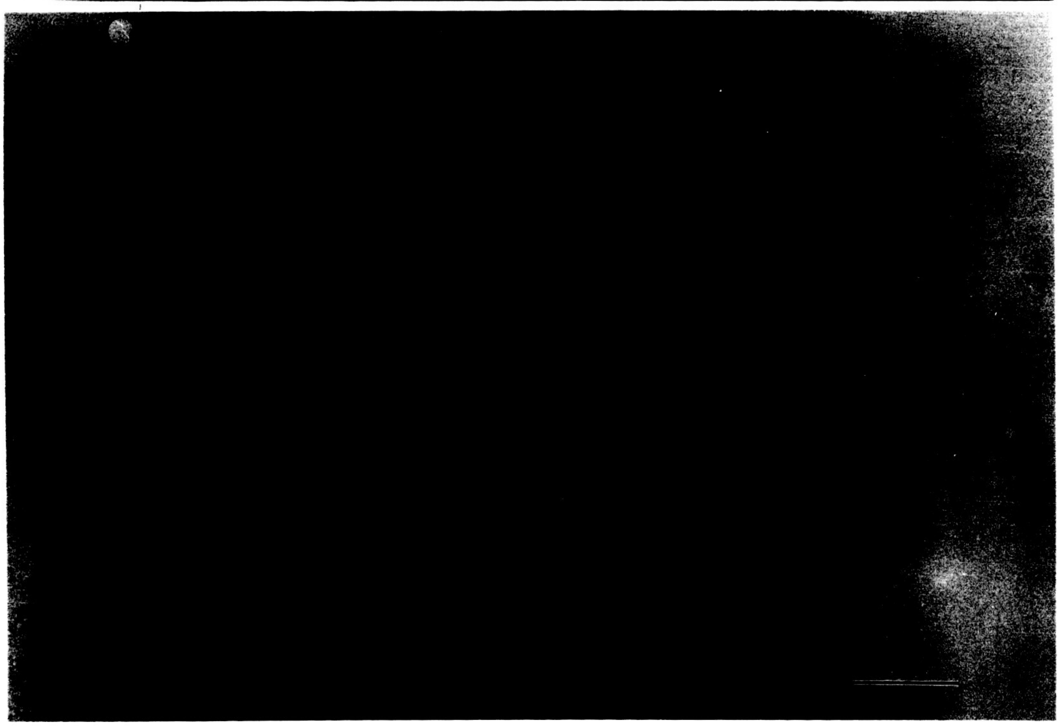
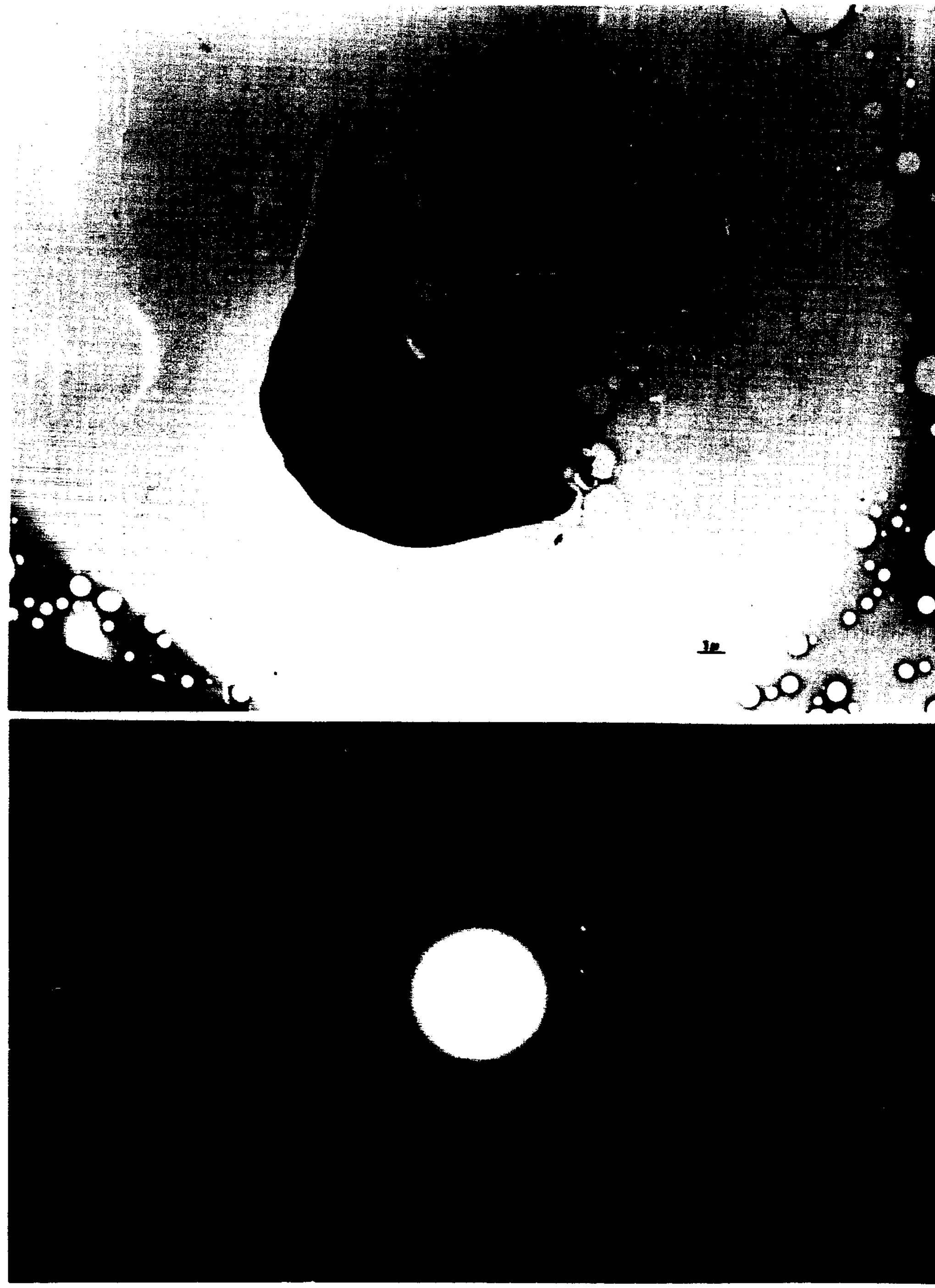


FIGURE C-2: ELECTRON MICROGRAPHS OF REPRESENTATIVE CONTROL THIN FILMS OF FORMVAR COATED WITH CARBON AND SILICON MONOXIDE. ("Ames" Control Series No. E-23). THESE MICROGRAPHS WERE TAKEN FROM A LARGE SERIES OF PRINTS SYSTEMATICALLY RECORDED AT HIGHER ELECTRON OPTICAL MAGNIFICATIONS TO MAP OUT THE ENTIRE VISIBLE SPECIMEN FILM AREA IN EACH CONTROL SAMPLE. THE FILM SURFACES ARE VERY SMOOTH AND CONSISTENTLY FREE FROM ANY RECOGNIZABLE PARTICULATE OR DROPLET CONTAMINANTS,ESPECIALLY OF THE (ORGANIC) TYPE FREQUENTLY SEEN IN THE EXPOSED "LUSTER" SPECIMENS FEATURING A MARKED SENSITIVITY TO ELECTRON BEAM IRRADIATION. EXCEPT FOR OCCASIONAL "HOLES" VISIBLE IN THE UNDERLYING FORMVAR SUBSTRATE THESE FILMS ARE OF EXCEPTIONAL UNIFORMITY AND COHERENCE. NOTICE THE "CLEAN" MARGINS OF THE HOLES WHICH DO NOT SHOW THE TYPICAL DROPLET PATTERNS COMMONLY FOUND IN CERTAIN OF THE EXPOSED "LUSTER SPECIMENS" (SEE,FOR EXAMPLE, FIGURES L-13 to L-15). MICROGRAPHS RECORDED WITH LOW INTENSITY MICROBEAM ILLUMINATION AND SPECIAL COLD STAGE(LIQUID NITROGEN,TYPE ARMBRUSTER) UNDER CONTROLLED CONDITIONS GIVING A REDUCED CONTAMINATION RATE OF ABOUT 0.1 Å PER SECOND IN ELMISKOP IA FITTED WITH "VITON" GASKETS. EXPOSURES ON 70 mm KODAK HIGH DEFINITION FILM OR ON KODALITH PHOTOGRAPHIC FILM PERMITTING UNINTERRUPTED RECORDING OF 70 to 90 FRAMES WITHOUT BREAKING THE HIGH VACUUM,THUS FURTHER REDUCING CONTAMINATION PROBLEMS. MAGNIFICATIONS:(in reproductions): 12,000 X.



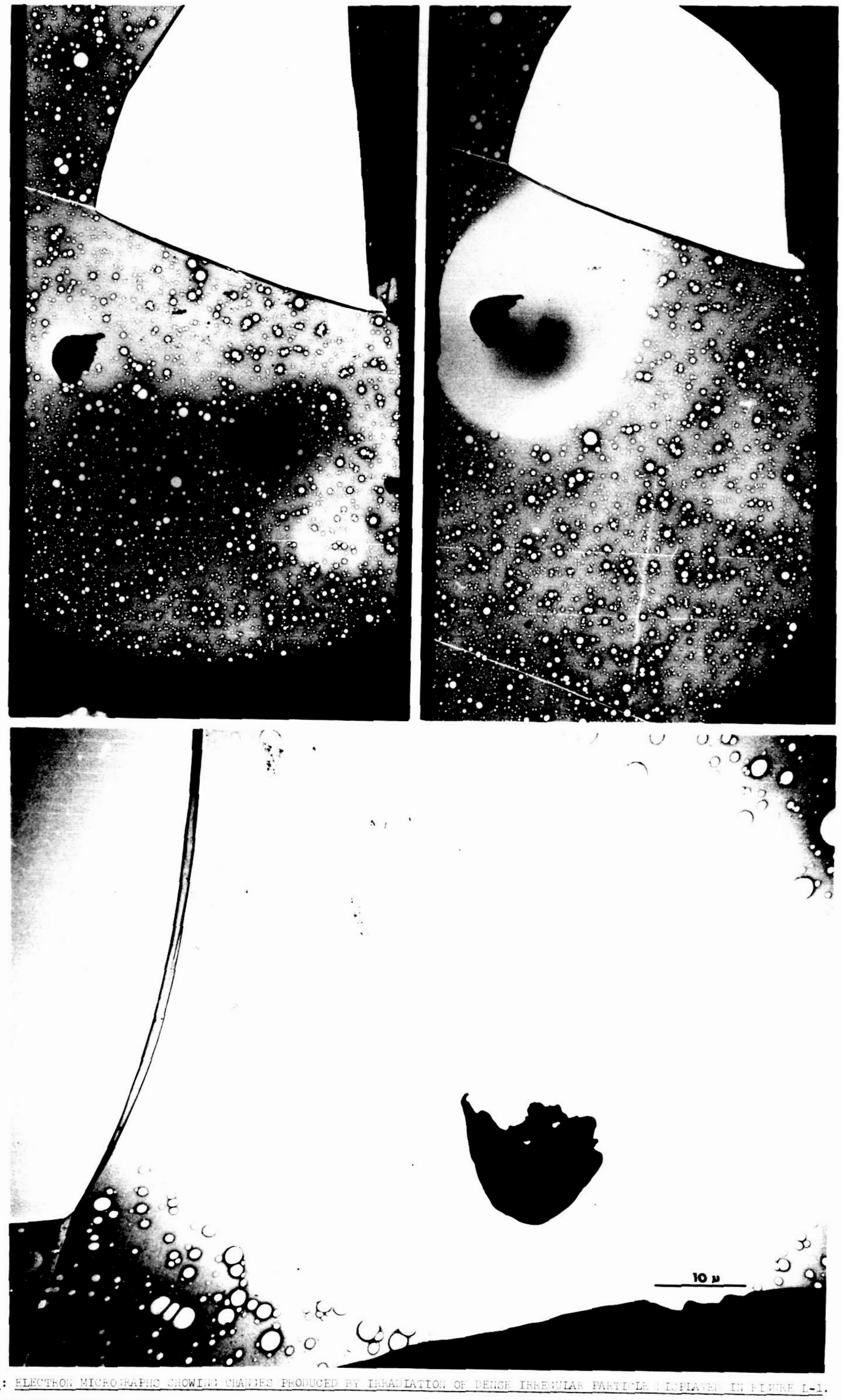
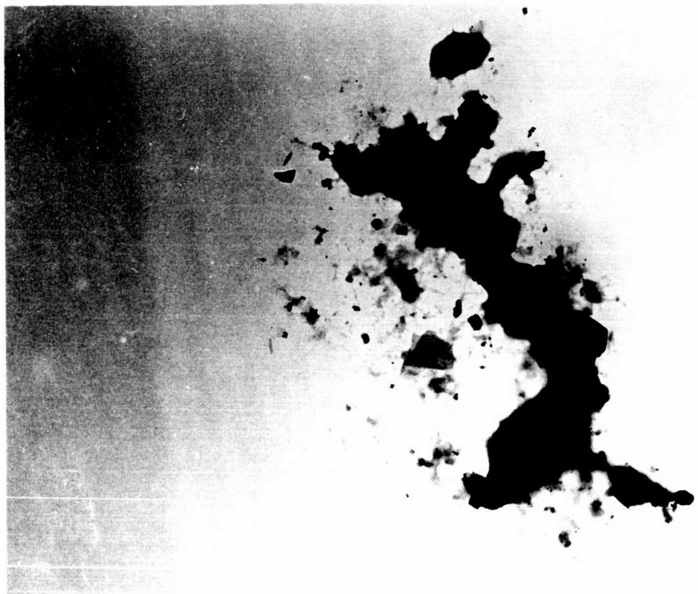


FIGURE L-2: ELECTRON MICROGRAPHS SHOWING CHANGES PRODUCED BY IRRADIATION OF DENSE IRREGULAR PARTICLE DISPLAYED IN FIGURE L-1. THE SERIES OF CHARACTERISTIC CHANGES RECORDED IN THIS SERIES OF MICROGRAPHS OCCURRED WITHIN A FEW SECONDS AFTER INCREASING SHARPLY THE IMAGE CURRENT DENSITY OF THE ILLUMINATING ELECTRON MICROBEAM. THE AREA ADJACENT TO THE PARTICLE, WHICH ALREADY EXHIBITED SIGNS OF CRACKING AND PUNCTURING OF THE FILM (Carbon film on Formvar) PROMPTLY RUPTURED, AND THE DENSE PARTICLE BEGINS TO "MELT" OR EVAPORATE, ACCOMPANIED BY THE FORMATION OF A LIGHT "HALO" AROUND THE PARTICLE. THIS MAY BE THE RESULT OF SELECTIVE REMOVAL OF SOME ELECTRON-SENSITIVE MATERIAL SURROUNDING THE PARTICLE. THE JAGGED, ANGULAR CONTOURS OF THE DENSE PARTICLE ARE OBLITERATED AND IT SHRINKS OR BECOMES MARKEDLY DISTORTED (bottom frame), FINALLY TEARING THE UNDERLYING FILM. THE CORE OF THE PARTICLE APPEARS TO BE QUITE REFRACTORY TO INTENSE ELECTRON BOMBARDMENT, AND THE TYPICAL EVAPORATION AND RECONDENSATION OF TINY DROPLETS DESCRIBED BY C.L. HEMENWAY AND R.K. SOBERMAN (Astronom. J. 67,2 4,19 2)(See also our Fig. M-1) WAS NOT OBSERVED. IF MORE PARTICLES OF THIS TYPE CAN BE FOUND IT MIGHT BE POSSIBLE TO TRY ULTRATHIN SECTIONING WITH THE MORAN DIAMOND KNIFE AND ULTRAMICROTOME (after appropriate embedding) IN ORDER TO OBTAIN BETTER PRESERVED SAMPLES FOR ELECTRON MICROSCOPY AND ELECTRON DIFFRACTION ANALYSIS. MAGNIFICATIONS: (red.): 500 X (top); 1200 X (bottom).



1µ

FIGURE L-3: ELECTRON MICROGRAPHS OF AGGREGATED DENSE-"FLUFFY" PARTICLES FOUND ON THIN FILM OF [illegible] EXPOSED DURING THE NOVEMBER 16, 19[illegible]5 LUSTER FLIGHT. ("LUSTER" SERIES No. L-3)

THIS VERY IRREGULAR TYPE IS SEEN TO CONSIST AT HIGHER MAGNIFICATION (frame below) OF NUMEROUS DENSE AND LIGHT PARTICLES, ABOUT 0.3 to 3 microns, ASSOCIATED WITH AN AMORPHOUS DIFFUSE MATERIAL WHICH SEEMS TO ENVELOP THE PARTICLES FORMING A LOOSE AGGREGATE. IN SOME RESPECTS IT RESEMBLES THE "FLUFFY" MICROMETEORITES FIRST DESCRIBED (Fig.10, p.[illegible]-259) BY HEMENWAY AND SOBERMAN. THE AMORPHOUS MATERIAL IS QUITE SENSITIVE TO (INTENSE) ELECTRON BOMBARDMENT AND EITHER EVAPORATES AWAY OR TRANSFORMS INTO FROTHY COMPLEXES (See Figs. L-13 to L-15). SOME OF THE ADJACENT THIN CRYSTALLINE LAMELLAE ARE UNDOUBTEDLY MICA CONTAMINANTS, BUT THE TYPICAL ASSOCIATION WITH THIS AMORPHOUS MATERIAL HAS NOT BEEN FOUND IN THE CONTROLS.

MAGNIFICATION: (in reproductions): 3700X - (top); 7400 X - (bottom).

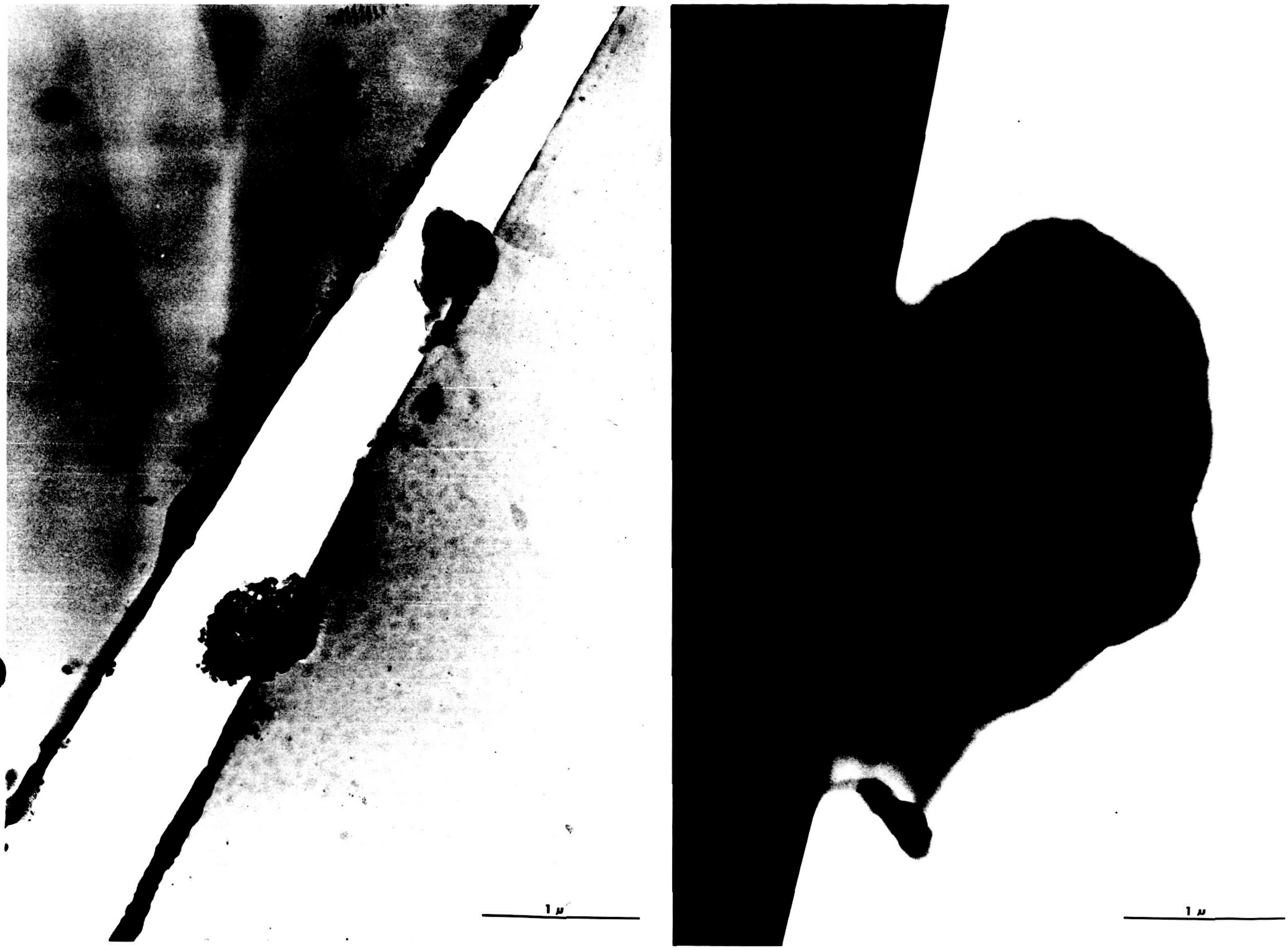


FIGURE L-4: ELECTRON MICROGRAPHS OF CONGLOMERATES OF SUBMICROSCOPIC DENSE PARTICLES FOUND AT THE EDGES OF RUPTURED THIN FILMS ON PLATINUM SPECIMEN HOLDERS EXPOSED DURING THE NOVEMBER 16, 1965 LUSTER FLIGHT TO COLLECT MICROMETEOROID DEBRIS DURING THE PEAK OF THE LEONIDS METEOR SHOWER. ("LUSTER" SERIES No. G-8).

THIS TYPE OF MINUTE DENSE PARTICLES (ABOUT 0.02μ to 0.2μ) IS FREQUENTLY FOUND IN VARIOUS STATES OF AGGREGATION AT THE EDGES OF CHARACTERISTIC TEARS AND FISSURES IN THE OTHERWISE RESISTANT FORMVAR-CARBON FILMS. THESE UNUSUAL TEARS RUNNING TRANSVERSELY ACROSS THE RIGID PLATINUM SLITS AND OFTEN INTERRUPTED BY ACTUAL "HOLES" PUNCHED THROUGH THE 400-500Å THICK FILMS ARE IN THEMSELVES NOTEWORTHY, SINCE THEY WERE NEVER FOUND IN THE CONTROLS, ARE UNLIKE ANY OTHER TYPE OF ACCIDENTAL FILM RUPTURES, AND ARE INVARIABLY ASSOCIATED WITH PARTICLES AND AMORPHOUS,ELECTRON-SENSITIVE MATERIAL (NOTE THICKENED EDGES COATED WITH THIS COMPONENT).MOREOVER, SINCE EACH SPECIMEN GRID WAS CHECKED WITH THE LIGHT MICROSCOPE AND THE INTEGRITY OF THE FILM RECORDED PHOTOGRAPHICALLY PRIOR TO TRANSFER IN THE ULTRAHIGH VACUUM CONTAINERS (FIGS. 1-6),THE TYPICAL RUPTURES OCCURRING IN ONLY A LIMITED NUMBER OF PARTICLE-CONTAINING SPECIMEN SUPPORTS MUST BE REGARDED AS SIGNIFICANT EVIDENCE IN SUPPORT OF EXTRATERRESTRIAL ORIGIN. THE APPEARANCE OF "FLAPPED HOLES" OF DIFFERENT SIZES IS ALSO CONSISTENT WITH THE ASSUMPTION THAT WE ARE DEALING WITH LOW-VELOCITY COLLISIONS AS FIRST SUGGESTED BY HEMENWAY & SOBERMAN(p.260). LARGER DENSE PARTICLES (right frame)ARE LESS FREQUENTLY FOUND AT THE EDGES OF HOLES.MAGNIFICATIONS:(red):both 21,500 X.

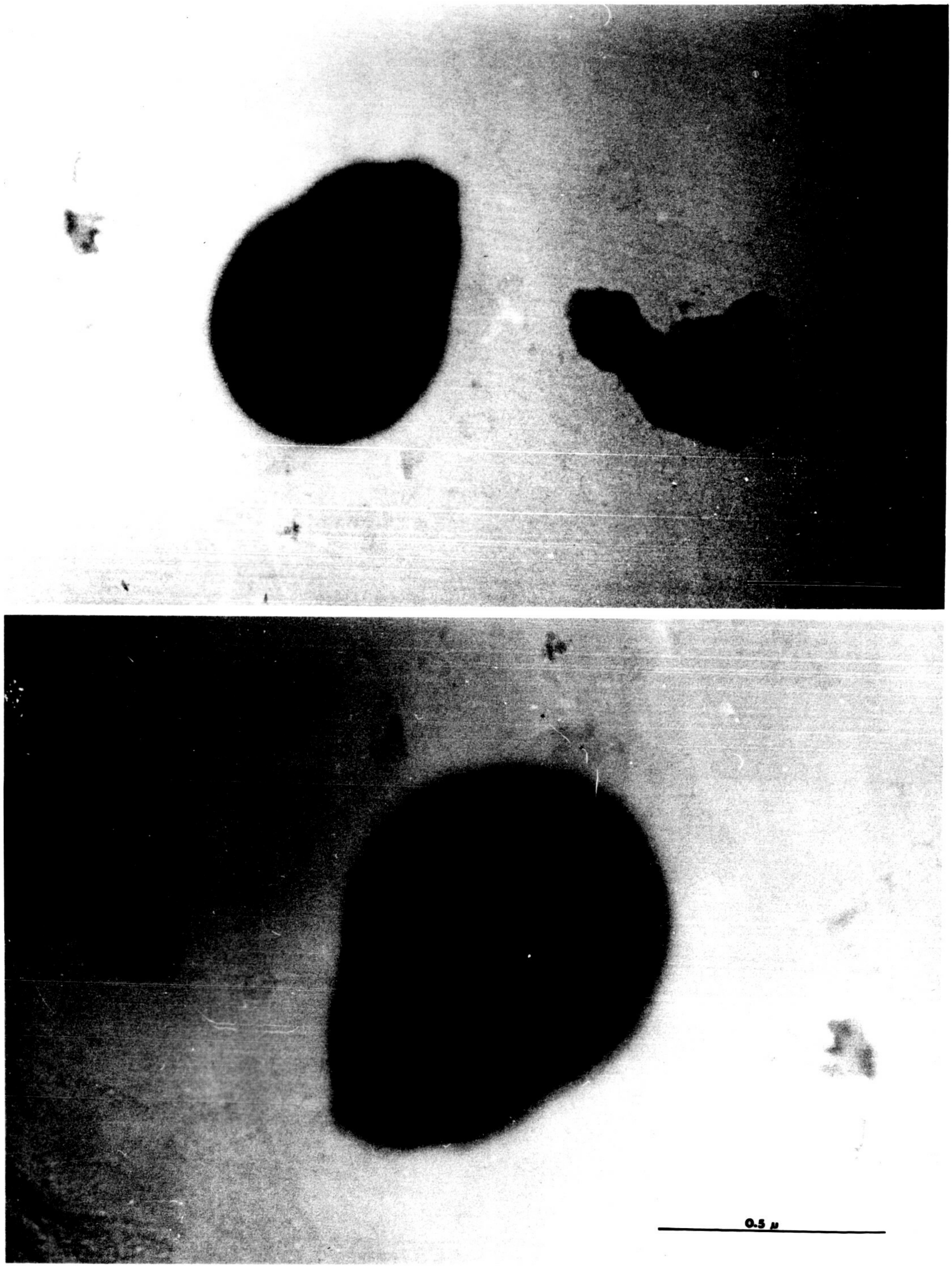


FIGURE L-5: ELECTRON MICROGRAPHS OF SUBMICROSCOPIC DENSE PARTICLES COATED WITH AMORPHOUS MATERIAL AS FOUND ON THIN FILM OF [illegible] SPECIMEN HOLDER EXPOSED DURING THE NOVEMBER 16, 1965 LUSTER FLIGHT. ("LUSTER" SERIAL No. [illegible]-25).

THIS TYPE OF SMALL (ABOUT 0.5μ to 0.[illegible]μ diameter) DENSE PARTICLES SURROUNDED BY A "HALO" OF AMORPHOUS MATERIAL (bottom frame) IS ALSO FOUND SINGLY, SCATTERED OVER CERTAIN FILM AREAS, BUT USUALLY NEAR A CRATERED REGION OR NOT FAR FROM OTHER PARTICULATE AGGREGATES. IT IS POSSIBLE TO DISTINGUISH ROUGHLY BETWEEN "ELECTRON DENSE" PARTICLES, AND "LIGHT PARTICLES", BUT BOTH SHARE THE CLOSE ASSOCIATION WITH THE AMORPHOUS MATERIAL WHICH IN MANY WAYS RESEMBLES THE ORGANIC (LIPID OR HYDROCARBON) COATINGS REGULARLY FOUND IN EM STUDIES OF PARTICLES PREPARED FROM THESE SYSTEMS. FURTHER ANALYSIS BY SELECTIVE EXTRACTION WITH ORGANIC SOLVENTS, OR VACUUM-DISTILLATION UNDER CONTROLLED CONDITIONS CAN NOT BE ADEQUATELY CARRIED OUT ON THE FORMVAR-CARBON FILMS OF THESE SPECIMEN HOLDERS, BUT THIS APPROACH IS QUITE FEASIBLE WHEN DEALING WITH SPECIMENS COLLECTED ON FRESHLY CLEAVED MICA SURFACES. EXPERIMENTS ALONG THESE LINES ARE ENVISAGED IN ATTEMPTS TO OBTAIN ADDITIONAL INFORMATION ON THE NATURE OF THIS AMORPHOUS COMPONENT.

MAGNIFICATIONS: (in reproductions): 40,000 X (top); [illegible]0,000 X (bottom).

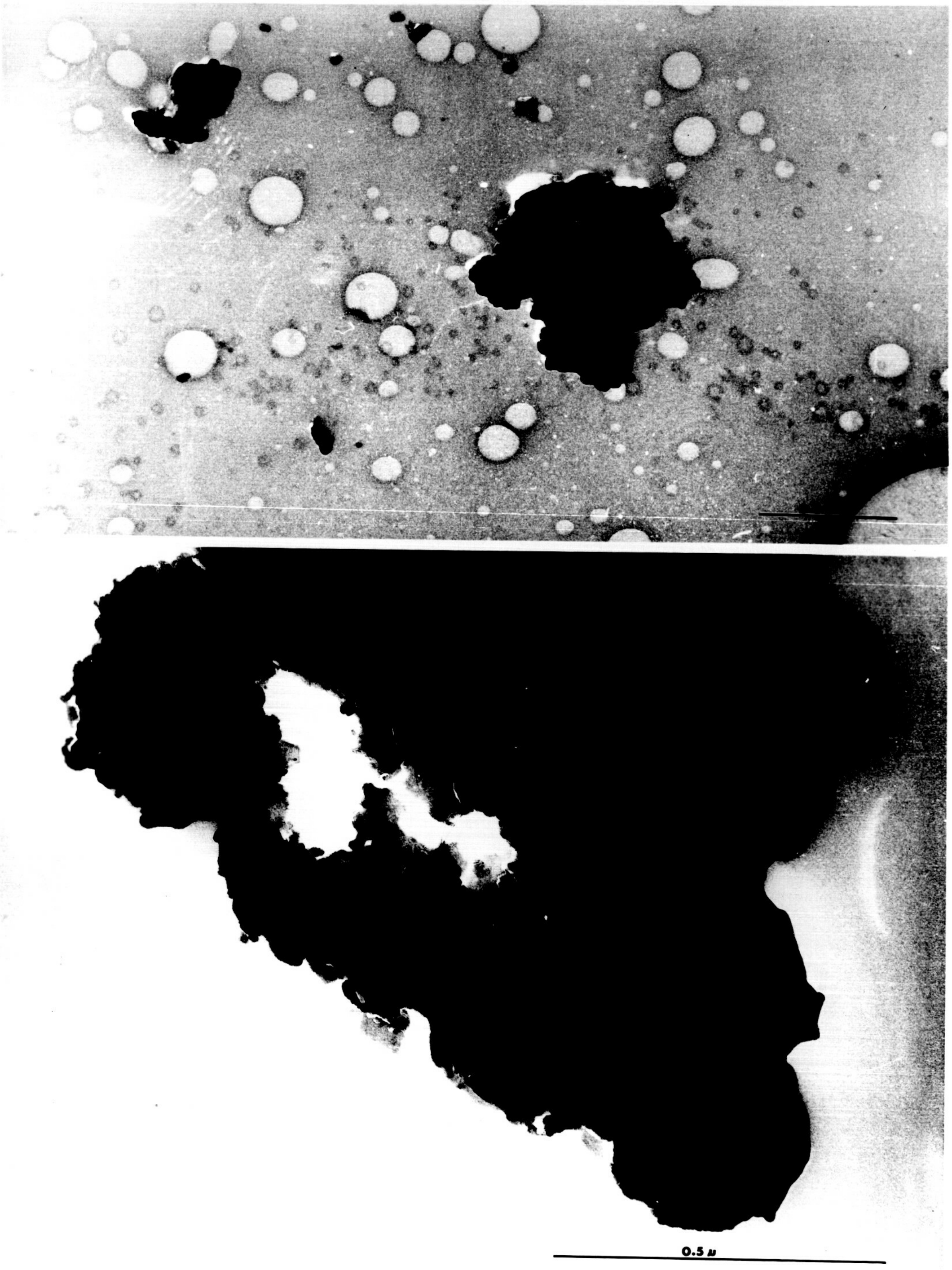


FIGURE L-[illegible]: ELECTRON MICROGRAPHS OF TERRESTRIAL (?) DENSE PARTICLES SHADOWED WITH CARBON AS FOUND ON THIN FILMS OF PLATINUM SPECIMEN HOLDER EXPOSED DURING THE NOVEMBER 1[illegible], 19[illegible] LUSTER FLIGHT. ("LUSTER" SERIES NO. [illegible]).

THESE PARTICLES HAVE BEEN SHADOWED WITH A THIN CARBON LAYER TO PROVIDE A PROTECTIVE COATING AGAINST THE EFFECTS OF INTENSE OR MODERATE ELECTRON BOMBARDMENT. THIS ALSO IMPARTS A THREE-DIMENSIONAL VIEW OF THE PARTICLE CONFIGURATION AND DISPLAYS SOME OF THE SURFACE FEATURES WHICH ARE USUALLY OBSCURED. THIS TYPE OF PARTICLE EXHIBITS A FRAGMENTED LAYERED STRUCTURE (SEE BOTTOM INSERT AT HIGHER ELECTRON OPTICAL MAGNIFICATION). SINCE PARTICLES SOMEWHAT SIMILAR TO THESE HAVE BEEN FOUND IN THE ("WHITE SAND") CONTROL SERIES AND THEY RESEMBLE CLAY PARTICLES IT IS TENTATIVELY ASSUMED THAT AT LEAST SOME OF THESE PARTICLES ARE TERRESTRIAL CONTAMINANTS. HOWEVER, FURTHER STUDIES ARE REQUIRED TO DIFFERENTIATE BETWEEN TRUE CONTAMINANTS AND POSSIBLE "SILICATE" EXTRATERRESTRIAL PARTICLES WHICH MIGHT HAVE BEEN COLLECTED IN THE COURSE OF THE LUSTER EXPERIMENT.

MAGNIFICATIONS: (in reproductions): 37,000 X (top); 90,000 X (bottom).

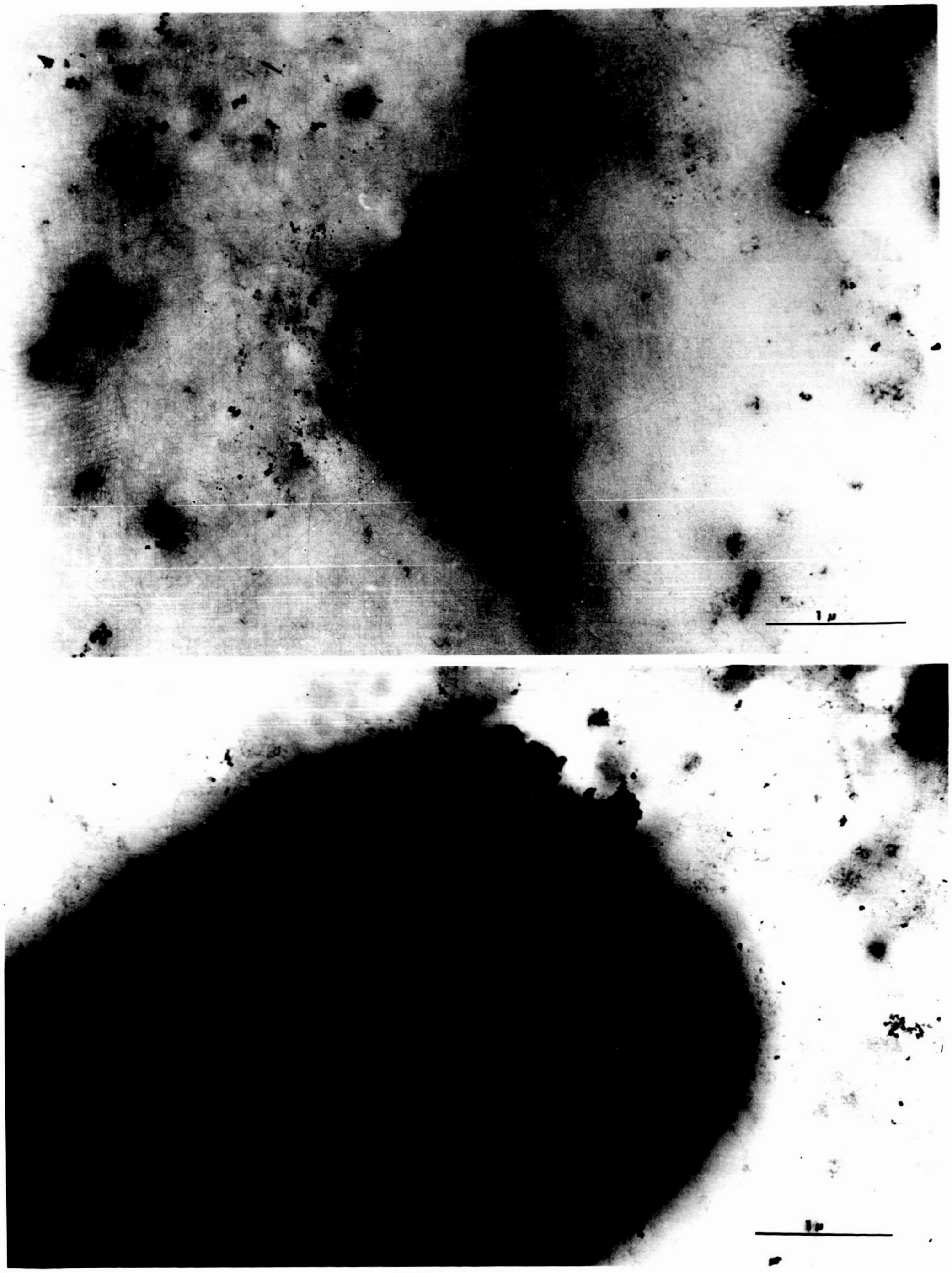
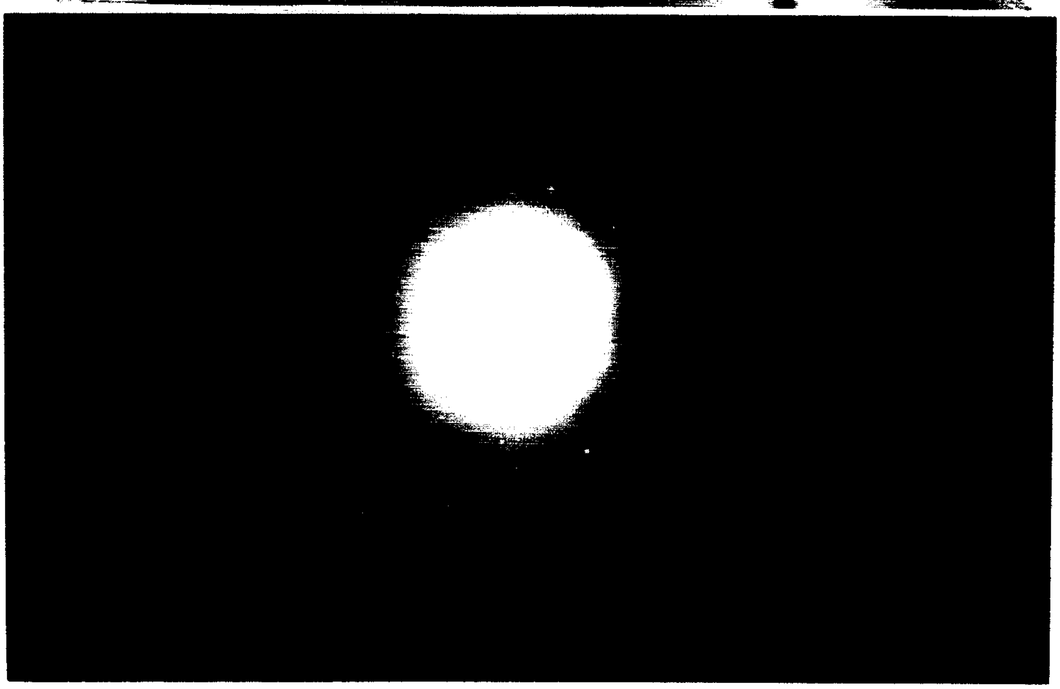


FIGURE L-7: ELECTRON MICROGRAPHS OF IRREGULAR FRAGMENTED DENSE PARTICLES COATED WITH AMORPHOUS MATERIAL AS FOUND ON THIN FILM OF PLATINUM SPECIMEN HOLDER EXPOSED DURING THE NOVEMBER 16, 1965 LUSTER FLIGHT. ("LUSTER" SERIES No. D-4)

THIS TYPE OF PARTICLE COMPLEX IS IN MANY RESPECTS SIMILAR TO THE MINUTE PARTICLE SYSTEMS FOUND AROUND THE EDGES OF FILM FISSURES AND HOLES. A DISTINGUISHING FEATURE OF THIS COMPARATIVELY FREQUENTLY OBSERVED PARTICLE COMPLEX IS THE ABUNDANCE OF THE AMORPHOUS MATERIAL AND EXTENSIVE DEGREE OF DISPERSION OF THE EMBEDDED DENSE PARTICLES MEASURING ONLY 200 Å IN THE LOWER SIZE RANGE TO ABOUT 5000 Å FOR THE LARGER PARTICULATE COMPONENTS (top frame). THE PREPONDRANCE OF THE AMORPHOUS MATERIAL SOMETIMES OBSCURES THE BOUNDARIES OF THE INDIVIDUAL CONSTITUENT PARTICLES (bottom frame) AND GIVES THE IMPRESSION THAT WE ARE DEALING WITH MUCH LARGER PARTICLES. PATCHES OF THIS MATERIAL ARE ENCOUNTERED IN ABOUT ONE THIRD OF ALL SPECIMEN HOLDERS EXAMINED IN OUR PRELIMINARY SURVEY. HOWEVER,THEY ARE PARTICULARLY PROMINENT IN FILMS WITH HOLES AND FISSURES,INDICATING THAT THESE PARTICLE COMPLEXES MIGHT POSSIBLY BE THE "DEBRIS" OR FRAGMENTED REMNANTS OF LARGER IMPINGING PARTICLES.THIS PARTICLE COMPLEX CAN ONLY BE DETECTED IN THE FORM SHOWN ABOVE IF SPECIAL PRECAUTIONS ARE TAKEN TO REDUCE ELECTRON BOMBARDMENT, CONSIDERABLY REDUCE CONTAMINATION EFFECTS AND PROTECT THE SPECIMEN DURING OBSERVATION IN THE ELECTRON MICROSCOPE BY USING LIQUID NITROGEN SPECIMEN COOLING DEVICES.MAGNIFICATIONS:(reprod.):both 19,000 X.

FIGURE L-8 : ELECTRON MICROGRAPH AND ELECTRON DIFFRACTION PATTERN OF IRREGULAR DENSE SUBMICROSCOPIC PARTICLES ASSOCIATED WITH AMORPHOUS MATERIAL AS FOUND ON THIN FILM OF PLATINUM SPECIMEN HOLDER EXPOSED DURING THE NOVEMBER 16, 1965 LUSTER FLIGHT. ("LUSTER" SERIES No. D-4).

THE TYPICAL ASSOCIATION OF THE DENSE AMORPHOUS MATERIAL WITH THE EXTREMELY SMALL (MINUTE PARTICLES ONLY 100 Å IN DIAMETER ARE PREDOMINANT IN MANY AREAS) DENSE PARTICLES IS SHOWN IN THE UPPER MICROGRAPH. BELOW IS A SELECTED AREA ELECTRON DIFFRACTION PATTERN FROM A SIMILAR REGION FEATURING A PATTERN OF CONCENTRIC SHARP RINGS WITH DISCRETE SPOT PATTERNS ASCRIBED TO THE (CRYSTALLINE) PARTICULATE COMPONENTS. PRECISE MEASUREMENTS ARE NOW BEING CARRIED OUT ON THESE DIFFRACTION PATTERNS AND ATTEMPTS WILL BE MADE TO DETERMINE THE LATTICE PARAMETERS OF THE DIFFRACTING MATERIAL. A CHARACTERISTIC SMALL-ANGLE PATTERN (reflections of ca. 8 Å to 17 Å) (See Fig. L-12) CAN ALSO BE RECORDED UNDER APPROPRIATE CONDITIONS FROM THESE SPECIMEN AREAS. MAGNIFICATION (in reproductions):19,000 X.



[illegible]

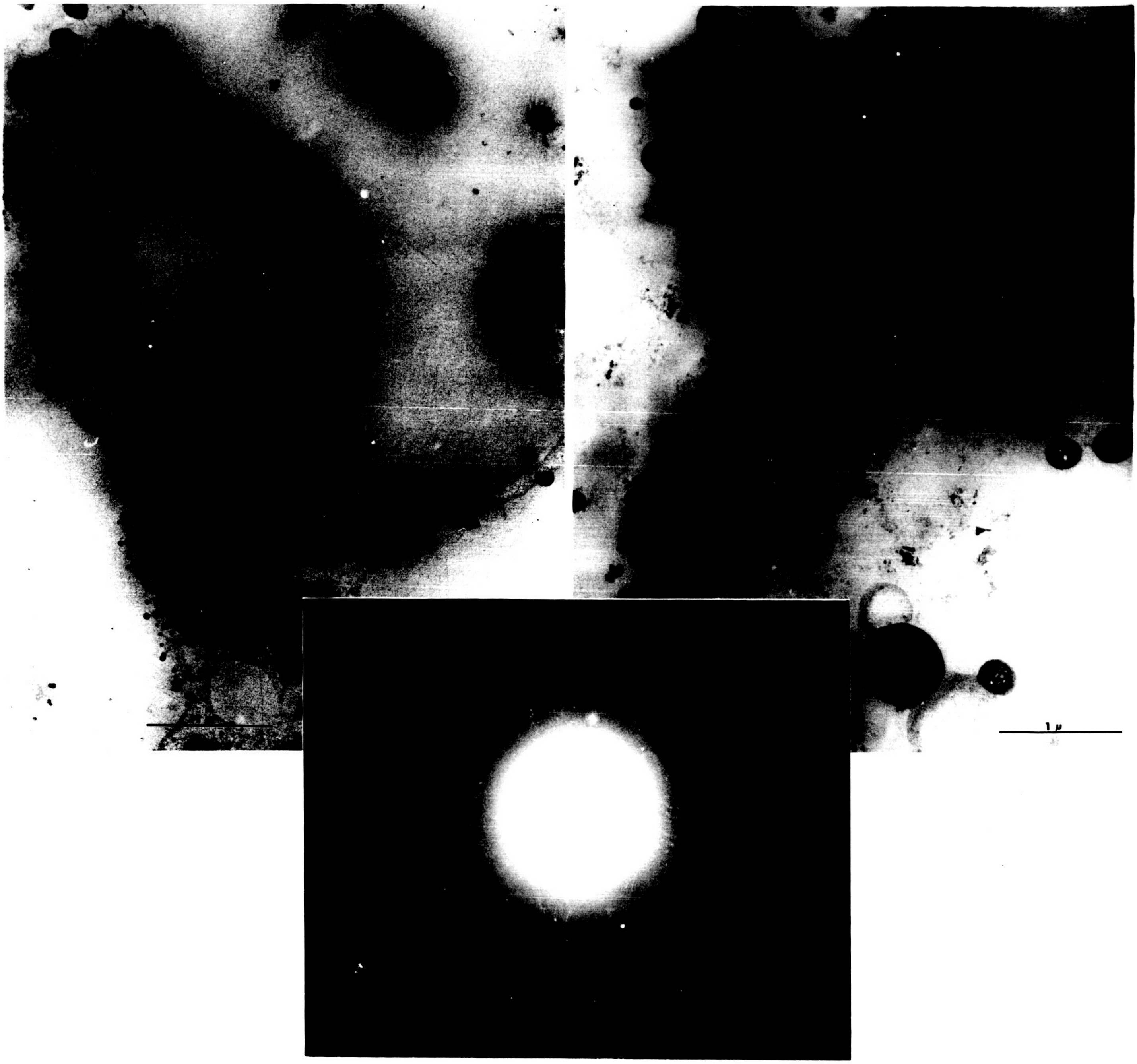


FIGURE L -10: ELECTRON MICROGRAPHS AND ELECTRON DIFFRACTION PATTERN FROM AREAS WITH DENSE SUBMICROSCOPIC DROPLETS [illegible] FILM OF PLATINUM SPECIMEN HOLDER EXPOSED DURING THE NOVEMBER 1[illegible], 19[illegible] LUSTER FLIGHT. ("LUSTER" SERIES No. [illegible]-17)
IN THE MICROGRAPH ON THE LEFT THE INDIVIDUAL (LARGE) DROPLETS ARE STILL WELL PRESERVED BEFORE ONSET OF THE [illegible] IMAGE DENSITY REQUIRED NORMALLY TO CLEARLY VISUALIZE THE SPECIMEN ON THE FLUORESCENT SCREEN. AS SHOWN IN THE [illegible] (top right) THIS SLIGHT INCREASE IN ELECTRON BOMBARDMENT SUFFICES TO COMPLETELY MODIFY THE [illegible] DROPLETS WHICH ARE [illegible] OR BLOW UP INTO A FOAMY AGGREGATE OF EMPTY BUBBLES. THE CORRESPONDING SELECTED AREA ELECTRON DIFFRACTION PATTERNS ARE [illegible] TO RECORD, BECAUSE THE REQUISITE FOCUSING OF THE MICROBEAM ON THE DROPLETS ALMOST IMMEDIATELY OBLITERATES THE PATTERN. [illegible] PRECAUTIONS,HOWEVER, A FEW USEFUL SMALL-ANGLE PATTERNS (SEE FIGS. L-11,L-1[illegible]) HAVE BEEN OBTAINED,WHICH ARE [illegible].
MAGNIFICATIONS:(in reproductions): both: 19,000 X.

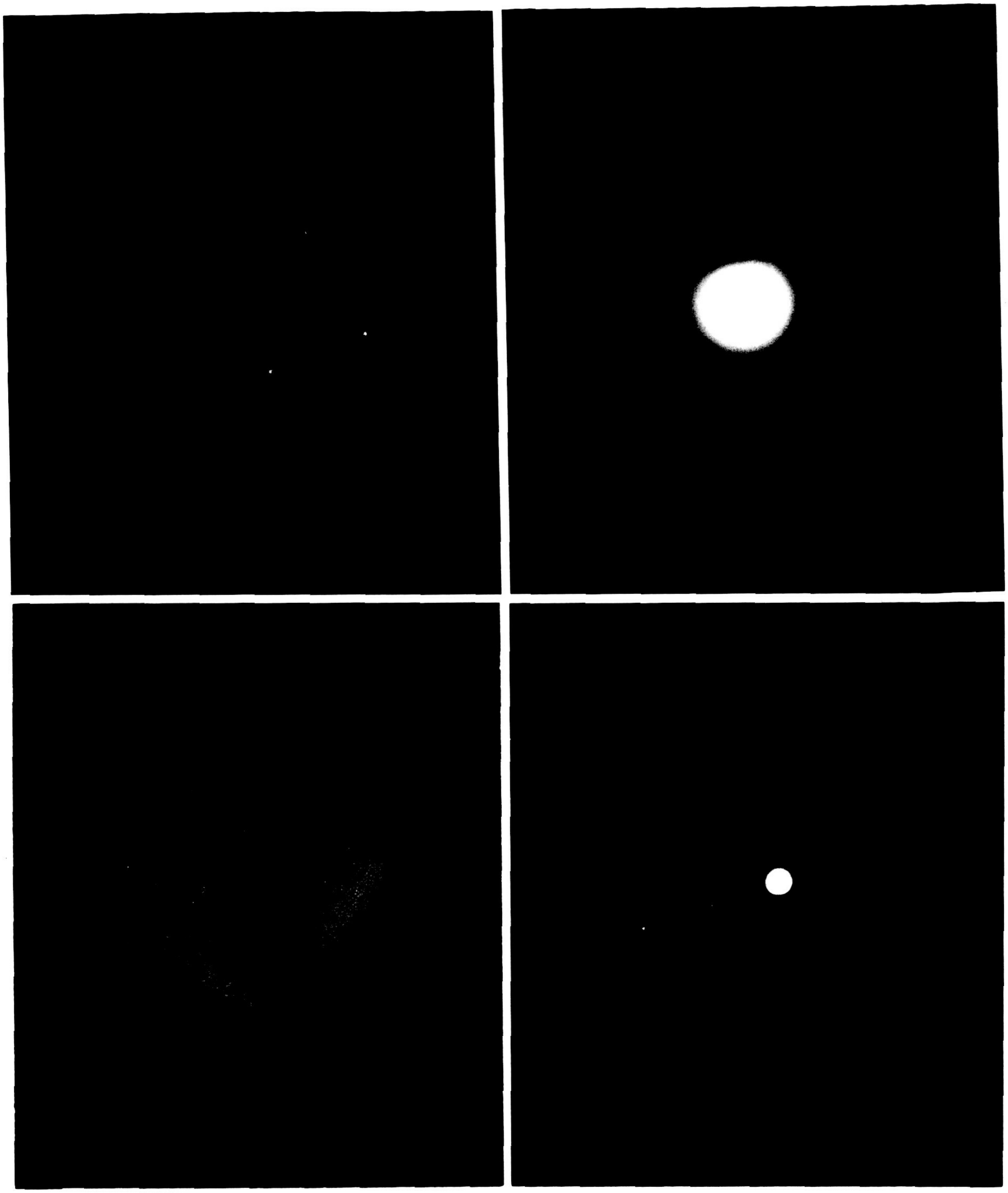


FIGURE L-11: ELECTRON MICROGRAPHS AND CORRESPONDING SELECTED-AREA ELECTRON DIFFRACTION PATTERNS RECORDED FROM DENSE SUBMICROSCOPIC DROPS FOUND CONDENSED ON THIN FILM OF PLATINUM SPECIMEN HOLDER EXPOSED DURING THE NOVEMBER 16, 1965 LUSTER FLIGHT. ("LUSTER"SERIES No.C-17) AS SHOWN IN THE ELECTRON MICROGRAPHS *ON THE LEFT SIDE, CERTAIN AREAS* CONTAINING THE TYPICAL (INTACT) DENSE SUBMICROSCOPIC DROPLETS HAVE BEEN MASKED OFF TO RECORD THE DIFFRACTION PATTERNS FROM THESE SITES ONLY. THE CORRESPONDING SELECTED-AREA, HIGH RESOLUTION (SMALL-ANGLE) PATTERNS (particularly bottom right showing enlarged segment of pattern close to central beam) FEATURE A NUMBER OF REFLECTIONS IN THE RANGE OF 7Å to about 18Å. HOWEVER, THESE PATTERNS ARE STILL NOT CONSISTENTLY REPRODUCIBLE, AND FURTHER IMPROVEMENTS IN RECORDING TECHNIQUES WITH SPECIMEN COOLING DOWN TO -180° COMBINED WITH IMAGE INTENSIFIERS OF THE HAINE-KOMODA TYPE WILL BE *REQUIRED IN ORDER TO OBTAIN* RELIABLE DATA ON THIS IMPORTANT COMPONENT. MAGNIFICATIONS: (in reprod.):(top left)19,000 X; 25,000X(bottom).

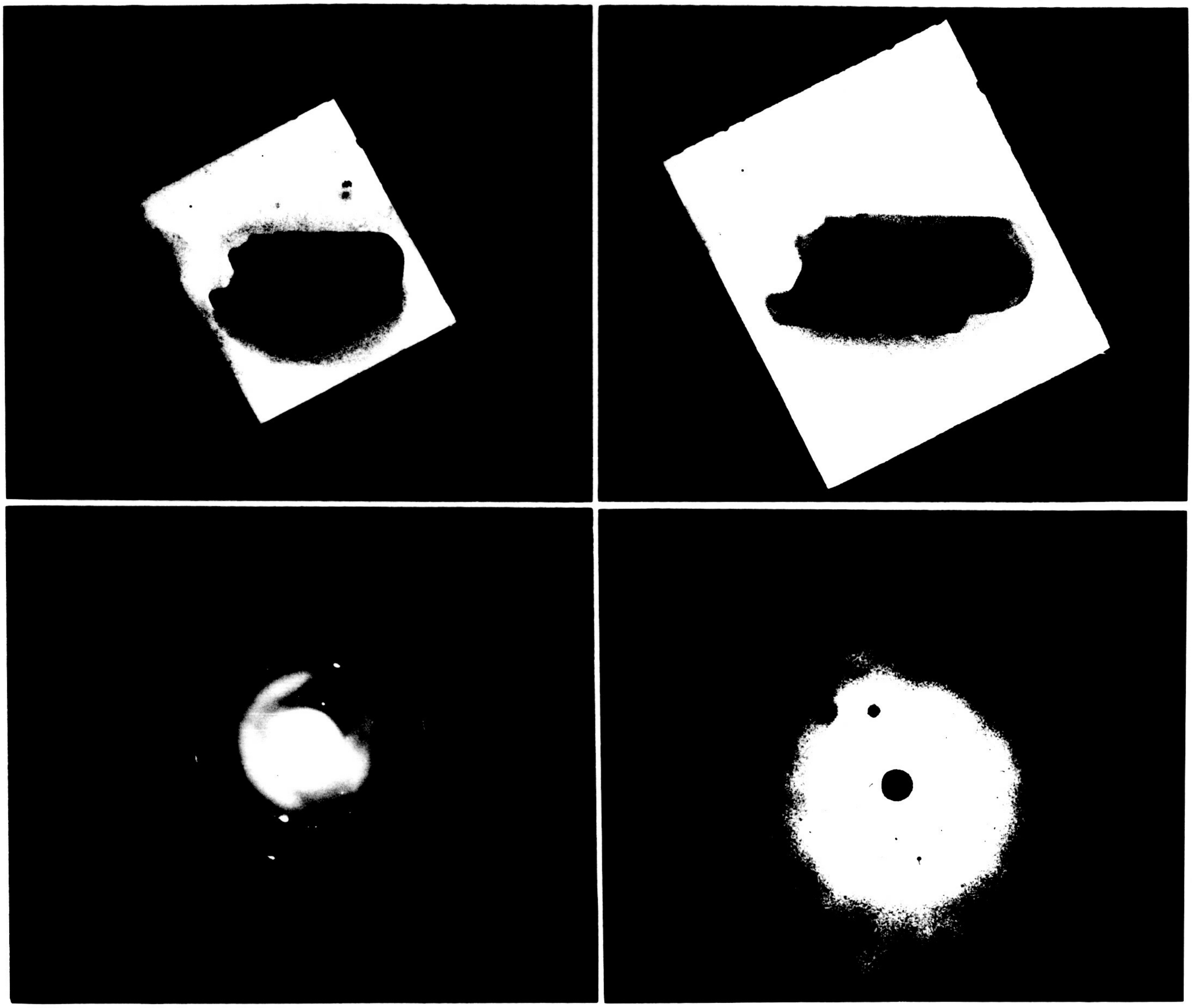
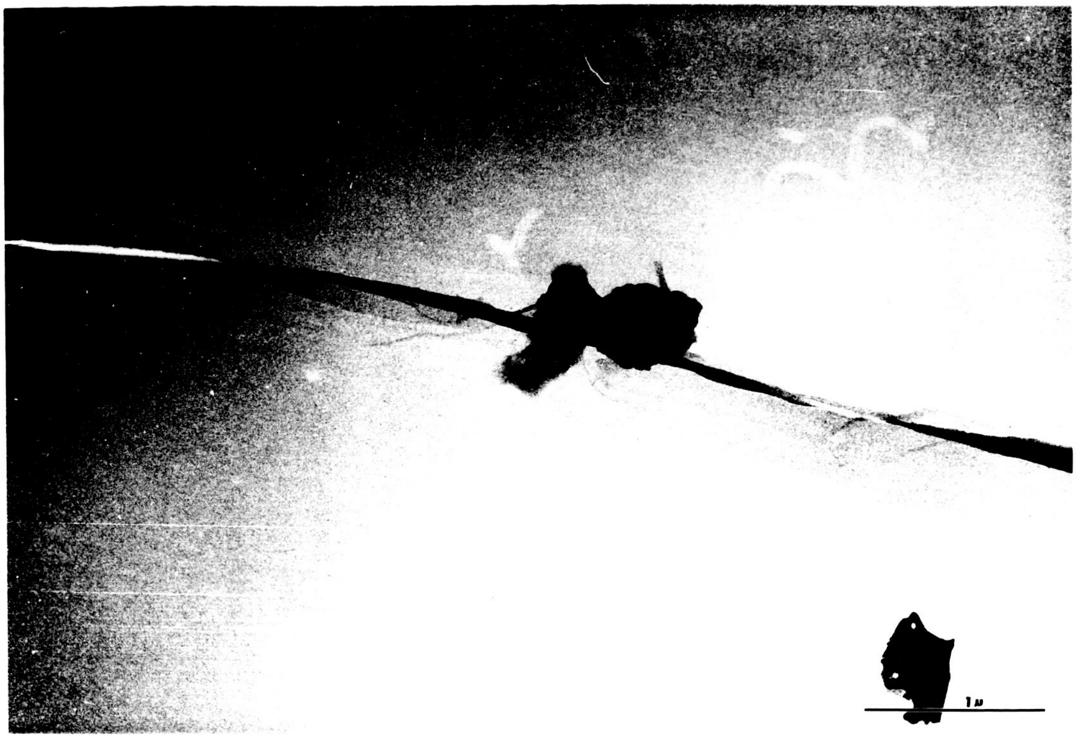


FIGURE L-12: ELECTRON MICROGRAPHS AND CORRESPONDING SELECTED-AREA ELECTRON DIFFRACTION PATTERNS [illegible] AMORPHOUS MATERIAL AND SUBMICROSCOPIC DROPLETS ASSOCIATED WITH PARTICLES AS FOUND ON THIN FILM OF [illegible] THE NOVEMBER 1, 1965 LUSTER FLIGHT. ("LUSTER"SERIES No. C-17)

WHEN THE PECULIAR AMORPHOUS MATERIAL IS FOUND (EITHER IN DROPLET FORM OR AS A COATING) IN CLOSE ASSOCIATION WITH (CRYSTALLINE OR AMORPHOUS) PARTICULATE COMPONENTS IT BECOMES POSSIBLE TO RECORD [illegible] DIFFRACTION PATTERNS USING THE [illegible] CRYSTALLINE DIFFRACTION PATTERNS AS A USEFUL GUIDE IN ALIGNING THE SYSTEM FOR RECORDING SMALL-ANGLE PATTERNS. AS SHOWN IN THESE TWO SERIES THE MICROGRAPHS OF THE MASKED PARTICLES SURROUNDED BY THE AMORPHOUS MATERIAL AND THE CORRESPONDING SELECTED-AREA HIGH RESOLUTION (SMALL-ANGLE) DIFFRACTION PATTERNS ARE BEING RECORDED AND EVALUATED FROM A NUMBER OF REPRESENTATIVE AREAS. IN ADDITION TO THE SINGLE-CRYSTAL DIFFRACTION PATTERNS (PROBABLY FROM MICA FLAKES) THERE ARE A SERIES OF DIFFUSE RINGS AND REFLECTIONS CLOSE TO THE CENTRAL BEAM (see particularly pattern bottom right,with enlarged central area) IN THE RANGE OF [illegible] Å TO ABOUT 18 Å. SOMEWHAT SIMIIAR PATTERNS HAVE BEEN OBTAINED FROM VARIOUS CYCLIC (e.g. cholesterol) AND ALIPHATIC (e.g.paraffins, fatty acids,etc.) COMPOUNDS. ATTEMPTS ARE CURRENTLY UNDERWAY TO IDENTIFY THE AMORPHOUS MATERIAL BY COMBINED SELECTIVE EXTRACTION EXPERIMENTS(e.g.with lipid solvents) AND PARTICULARLY BY DETAILED COMPARISON OF THESE ELECTRON DIFFRACTION PATTERNS WITH THE PATTERNS OF THE KNOWN ALIPHATIC AND AROMATIC HYDROCARBONS WHICH ARE BEING PRODUCED BY DR.E.ANDERS,R.HAYATSU,AND M.H.STUDIER AT THE UNIVERSITY OF CHICAGO AND ARGONNE NATIONAL LABORATORY THROUGH A FISCHER-TROPSCH TYPE REACTION BETWEEN CARBON MONOXIDE AND HYDROGEN USING IRON AND STONY METEORITES AS EFFECTIVE CATALYSTS. MAGNIFICATIONS:(reprod.):(top left):19,000X; (top,right): 25,000 X.



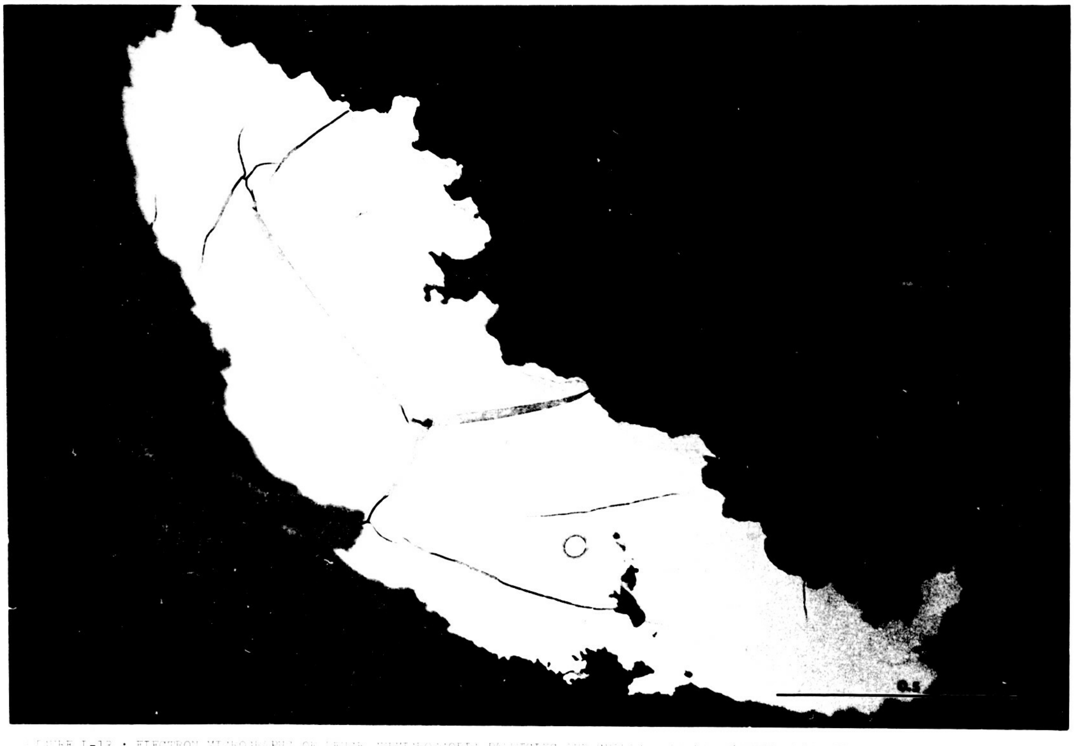


FIGURE I-13 : ELECTRON MICROGRAPHS OF DENSE MICROSCOPIC PARTICLES AND CHARA[illegible] OF THIN CARBON FILMS ON PLATINUM SPECIMEN HOLDERS EXPOSED DURING THE NOVEMBER 16, 1965 LUSTER FLIGHT [illegible] THE LEONIDS METEOR SHOWER. ("LUSTER" SPECIMEN NO. [illegible]).

THE ASYMMETRIC PARTICLES WITH DENSE CORE AND LIGHT COAT ARE FREQUENTLY FOUND SINGLY OR AGGREGATED AT THE [illegible] RUPTURES (top frame) IN THE OTHERWISE RESISTANT FORMVAR-CARBON FILMS. (SEE FIGS. I-4). IN ADDITION, THERE ARE CHARACTERISTIC RUPTURES IN THE (ca.400 Å thick) CARBON FILM (bottom frame) WHICH APPEAR TO CORRESPOND CLOSELY TO THE SILHOUETTE OF RATHER IRREGULAR PARTICLES. THESE CHARACTERISTIC RUPTURES, WHICH WERE NEVER FOUND IN THE CONTROLS, AND OCCUR IN ONLY A LIMITED NUMBER OF PARTICLE-[illegible] "LUSTER" SPECIMEN SURFACES, ARE CONSISTENT WITH THE ASSUMPTION THAT WE ARE DEALING WITH EXTRATERRESTRIAL PARTICLES IMPINGING ON THE THIN SPECIMEN FILMS AT RELATIVELY LOW VELOCITY. Magnification scales: 1μ (above); 0.5μ (below).

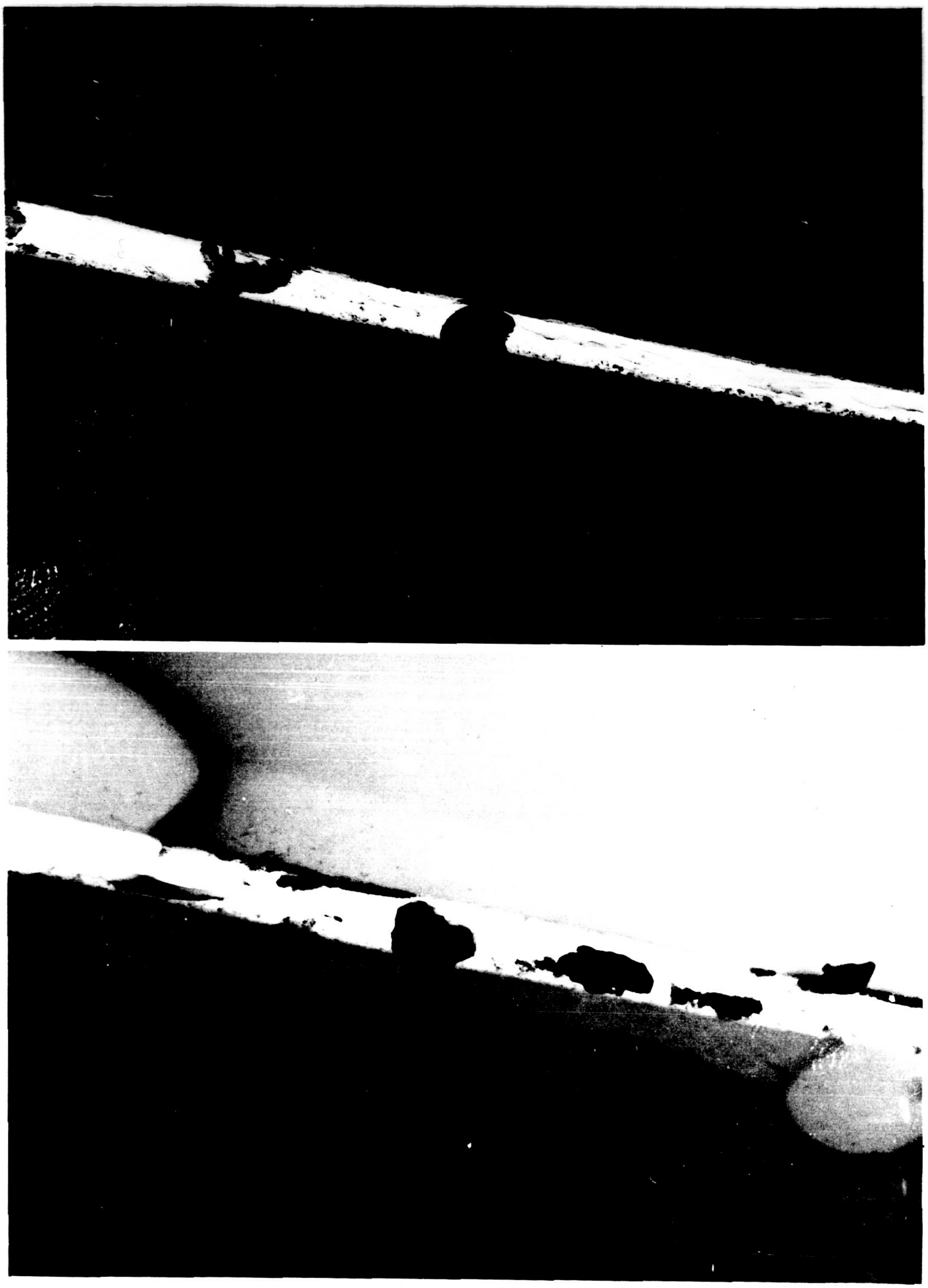
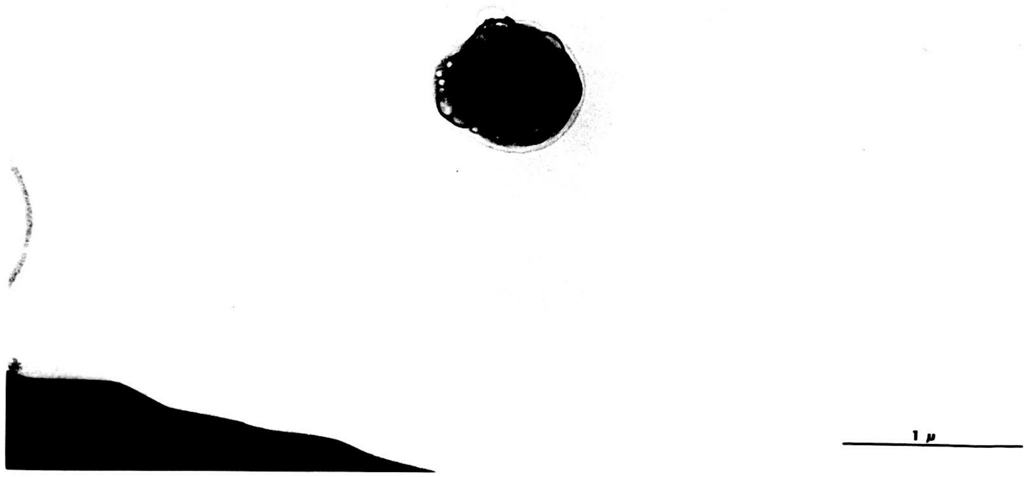


FIGURE L-14 : ELECTRON MICROGRAPHS OF CONGLOMERATES OF ASYMMETRIC DENSE PARTICLES FOUND AT THE EDGES OF RUPTURED THIN FILMS ON PLATINUM SPECIMEN HOLDERS EXPOSED DURING THE NOVEMBER 16, 1965 LUSTER FLIGHT. ("LUSTER" SERIES No. D-13).
THIS TYPE OF ASYMMETRIC DENSE PARTICLES (See also Figure L-4 & L-13) IS OFTEN FOUND IN DIFFERENT STATES OF AGGREGATION AT THE EDGES OF CHARACTERISTIC TEARS AND FISSURES IN THE CARBON-FORMVAR FILMS. THE PARTICLES FREQUENTLY FEATURE A ROUNDED "NOSE-CONE" CONFIGURATION (See also Figs. L-1,2) WHICH MAY BE RELATED TO THEIR MODE OF IMPINGEMENT ON THE RESISTANT THIN SPECIMEN SUBSTRATES. THESE PARTICLES OF ABOUT 0.1μ to 0.5μ ARE FREQUENTLY ENCOUNTERED IN MANY OF THE "LUSTER" SPECIMENS. PARTICLES OF THIS TYPE HAVE NOT YET BEEN DETECTED IN THE CONTROL SPECIMENS.



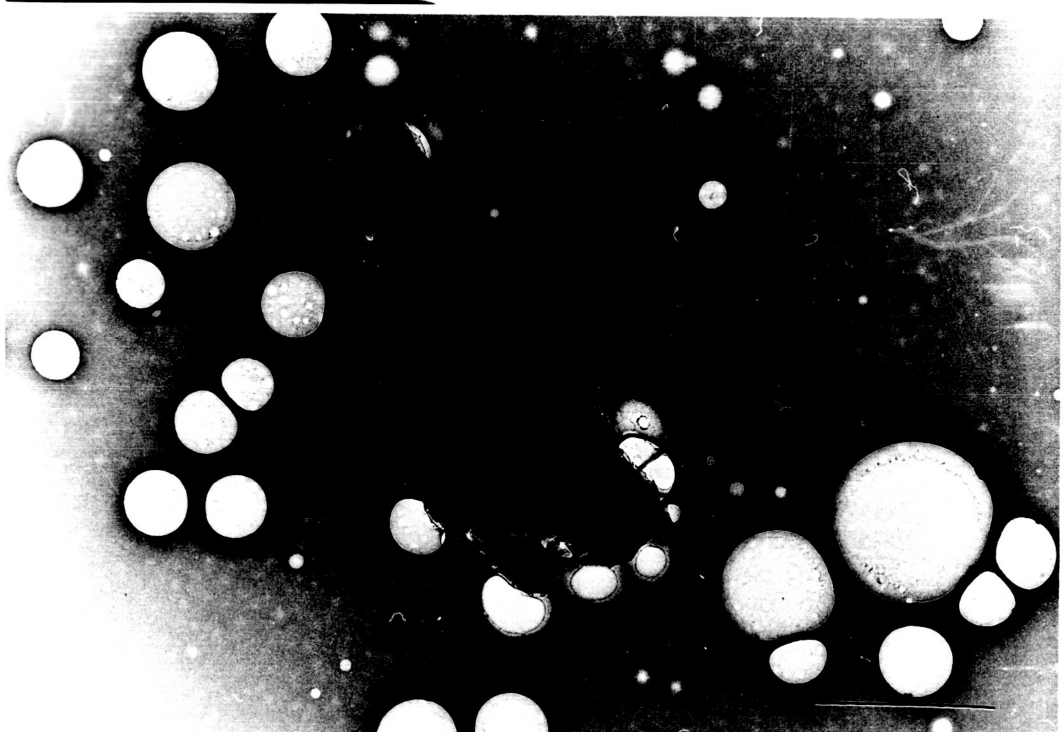


FIGURE [illegible]-15: ELECTRON MICROGRAPHS OF CHARACTERISTIC ELECTRON-SENSITIVE PARTICLES FOUND ON THIN FILMS OF PLATINUM SPECIMEN HOLDERS EXPOSED DURING THE NOVEMBER 16, 1965 LUSTER FLIGHT. ("LUSTER" SERIES No. [illegible]).

[illegible] PARTICLES (ABOUT 0.5μ to 3μ) FEATURES A DENSE CORE SURROUNDED BY A COATING OF AMORPHOUS MATERIAL WHICH IS VERY SENSITIVE [illegible] ELECTRON-BEAM IRRADIATION (bottom frame). THERE ARE ALSO SMALLER PARTICLES (top frame) WHICH APPEAR TO CONSIST [illegible] OF THIS ELECTRON-SENSITIVE MATERIAL. AFTER ELECTRON-BEAM BOMBARDMENT THEY EXPAND INTO CHARACTERISTIC "FOAMY" STRUCTURES.

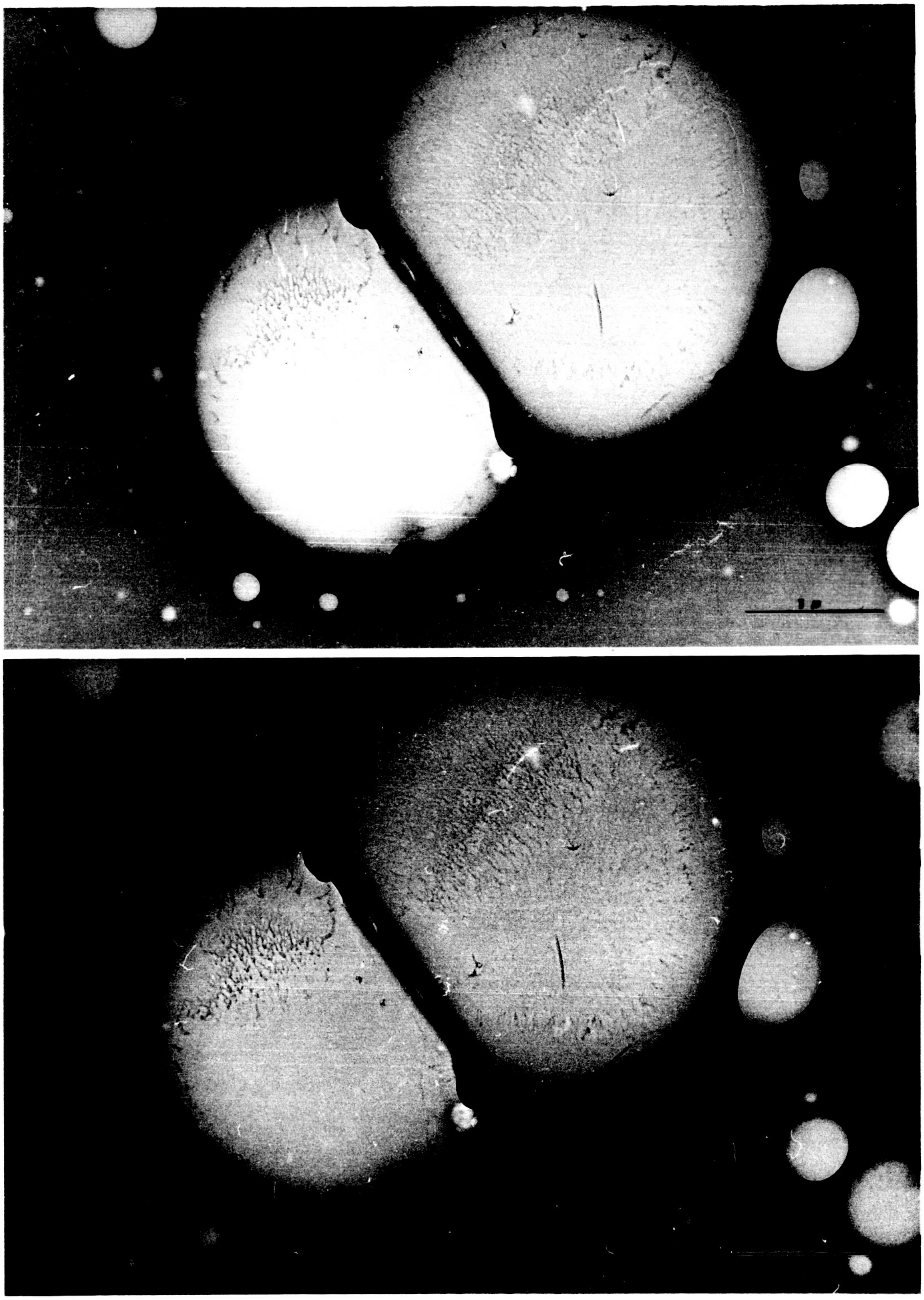
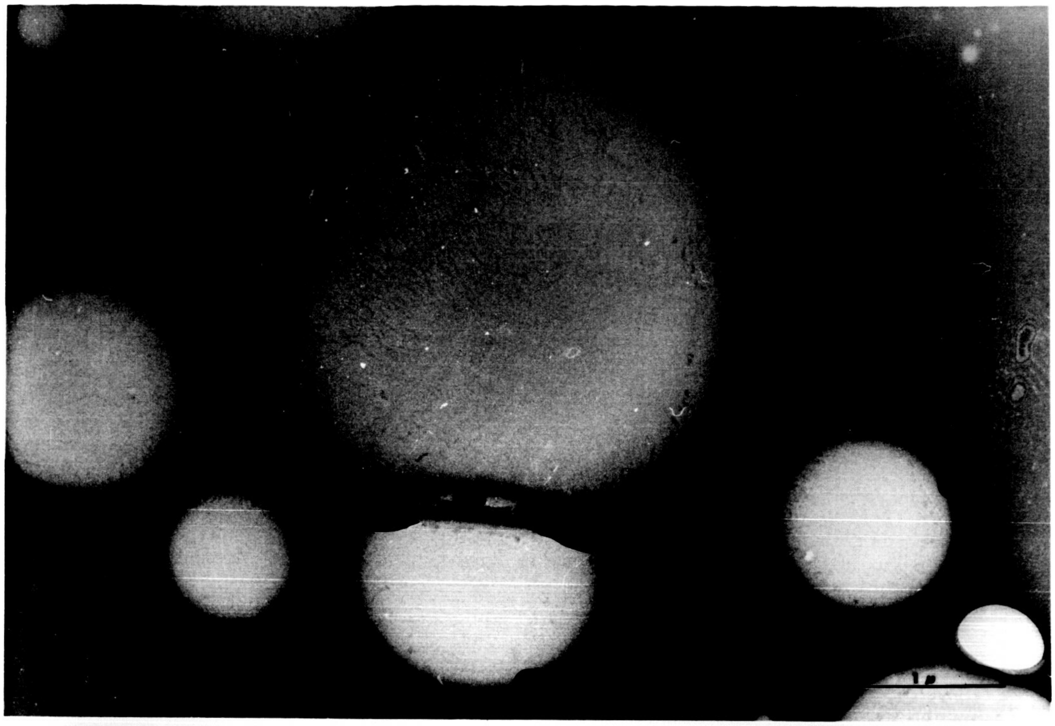


FIGURE L-16: COMPARATIVE ELECTRON MICROGRAPHS OF CHARACTERISTIC ELECTRON-SENSITIVE PARTICLES AND DROPLETS (BEFORE AND AFTER IRRADIATION) FOUND AT THE EDGES OF FENESTRATIONS IN THIN SPECIMEN FILMS EXPOSED DURING THE NOVEMBER 16, 1965 LUSTER FLIGHT. ("LUSTER" SERIES No. A-13).

THIS CHARACTERISTIC ELECTRON-SENSITIVE MATERIAL CLOSELY RESEMBLES THE "DROPLETS" AND AMORPHOUS MATERIAL FREQUENTLY FOUND IN THE "LUSTER" SPECIMENS (SEE FIGURES L-4-8). WHEN THESE AREAS (top frame) ARE EXAMINED WITH MICRO-BEAM ILLUMINATION OF LOW INTENSITY USING A LIQUID NITROGEN SPECIMEN COOLING DEVICE, THE DENSE DROPLETS ARE RELATIVELY WELL PRESERVED. HOWEVER, UPON SLIGHT INCREASE OF THE ELECTRON-BEAM INTENSITY, THE DROPLETS IMMEDIATELY VOLATILIZE AND TURN INTO TYPICAL "FOAMY" STRUCTURES (bottom frame). THIS MATERIAL, FREQUENTLY FOUND AT THE EDGES OF FENESTRATIONS IN THE CARBON-FORMVAR FILM, HAS NOT BEEN DETECTED IN THE CORRESPONDING CONTROL SPECIMENS. ORIGINAL MAGNIFICATIONS: 50,000 X.



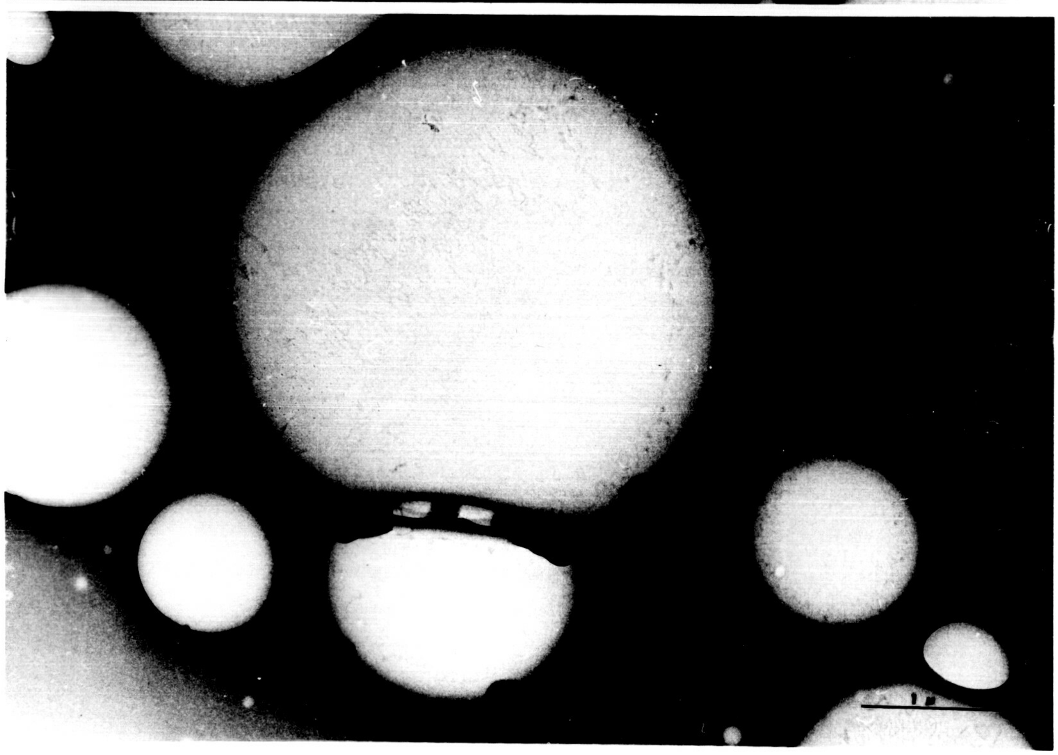
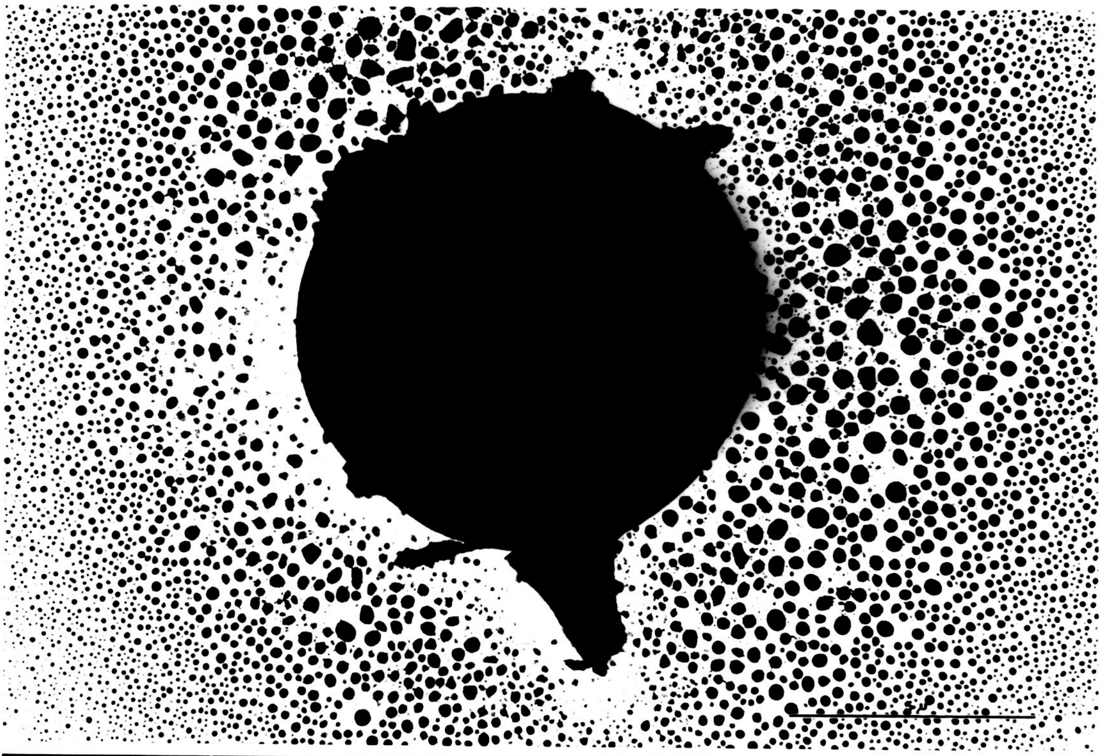
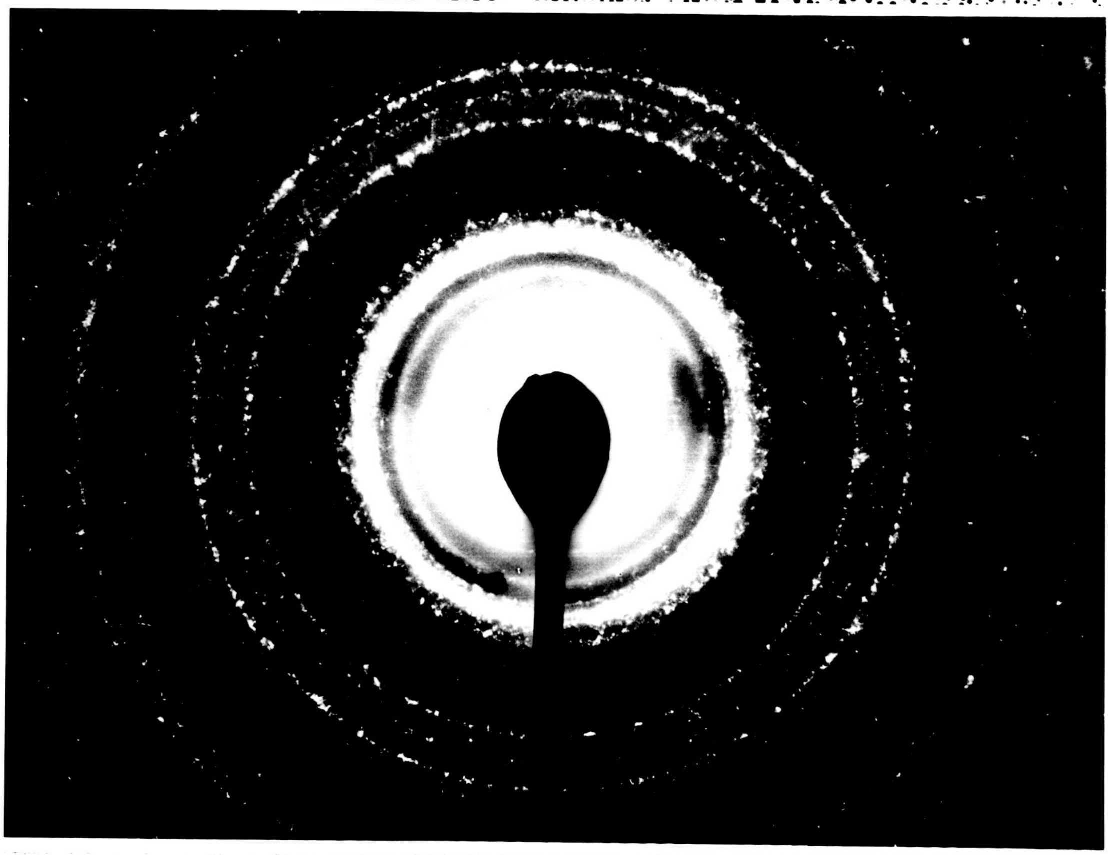


FIGURE L-17: COMPARATIVE ELECTRON MICROGRAPHS OF CHARACTERISTIC ELECTRON-SENSITIVE PARTICLES AND DROPLETS (BEFORE AND AFTER IRRADIATION) FOUND AT THE EDGES OF FENESTRATIONS IN THIN SPECIMEN FILMS EXPOSED DURING THE NOVEMBER 16, 1965 LUSTER FLIGHT. ("LUSTER" SERIES [illegible]).

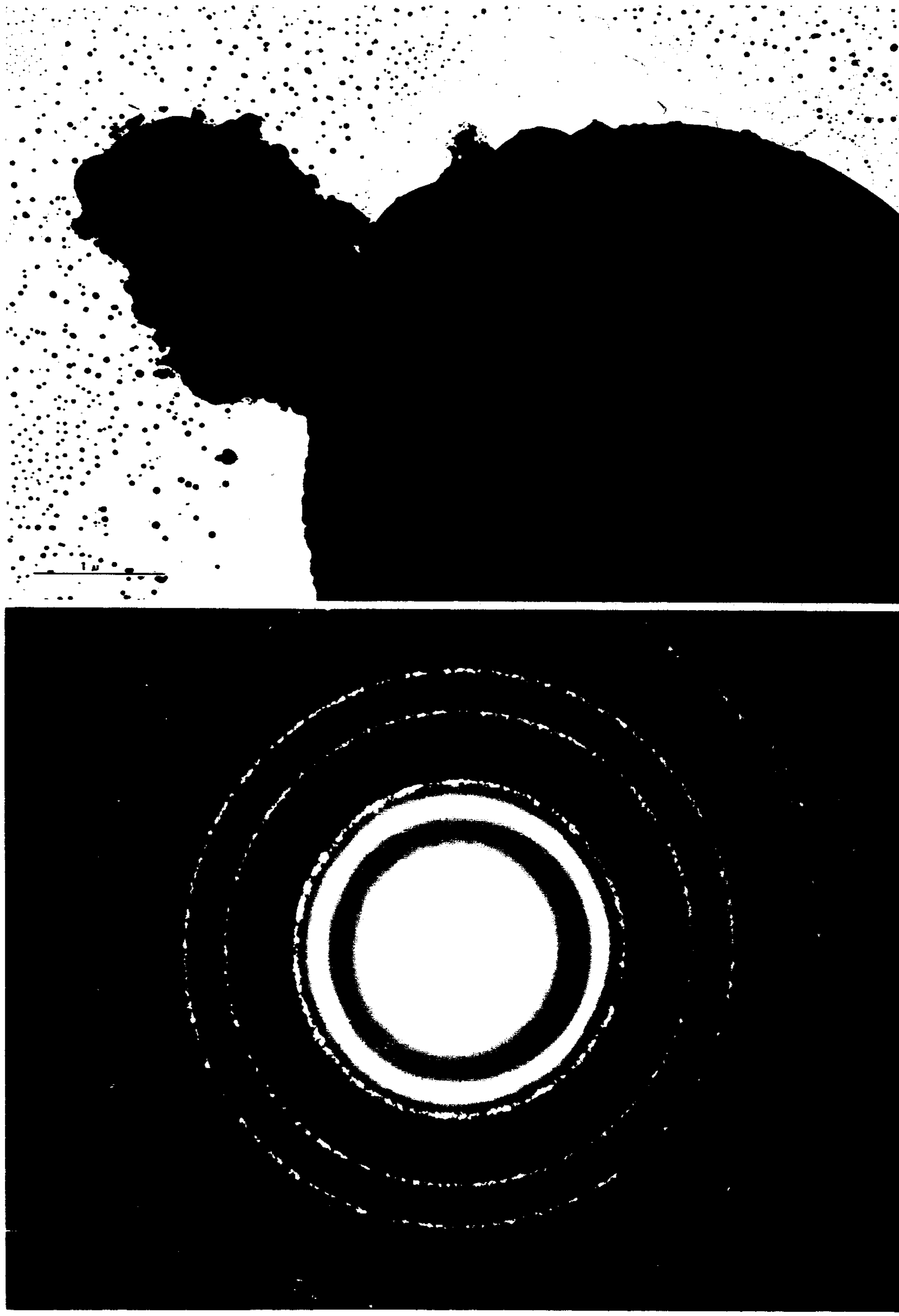
[illegible] MATERIAL CLOSELY RESEMBLES THE "DROPLETS" AND AMORPHOUS MATERIAL FREQUENTLY FOUND IN THE [illegible]. WHEN THESE AREAS ([illegible] frame) ARE EXAMINED WITH MICRO-BEAM ILLUMINATION [illegible] THE [illegible] ARE RELATIVELY WELL PRESERVED. HOWEVER, UPON SLIGHT INCREASE [illegible] "FOAMY" [illegible] (bottom frame). [illegible] FILM. [illegible] MAGNIFICATION: [illegible] X.

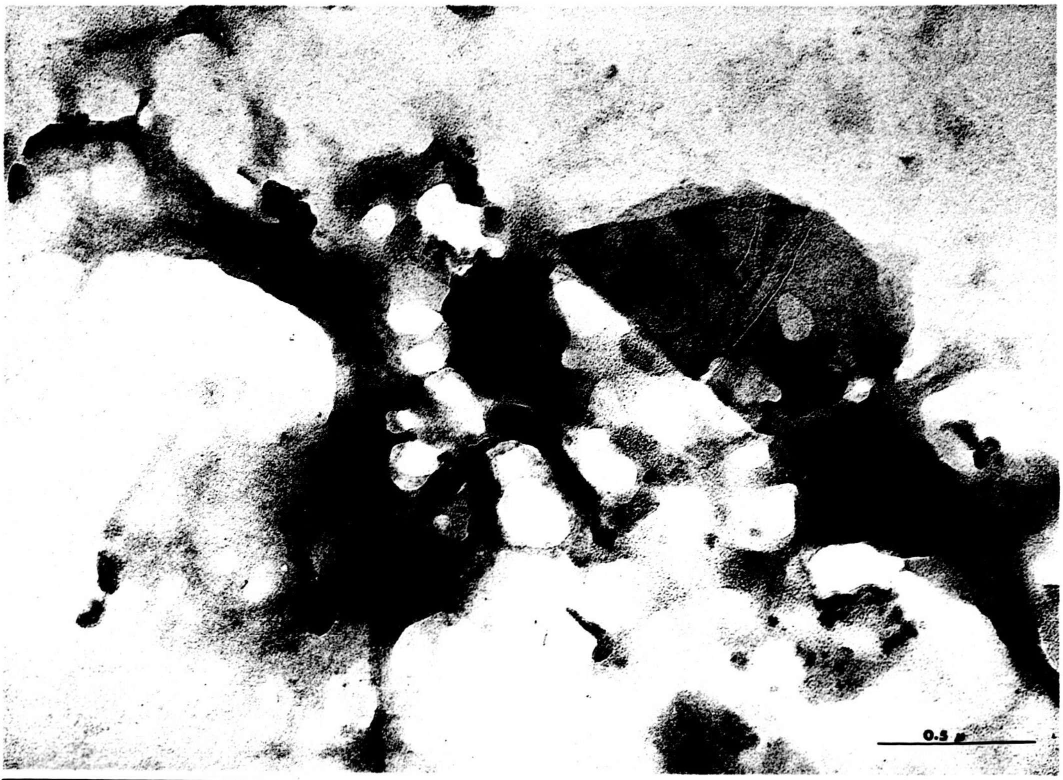


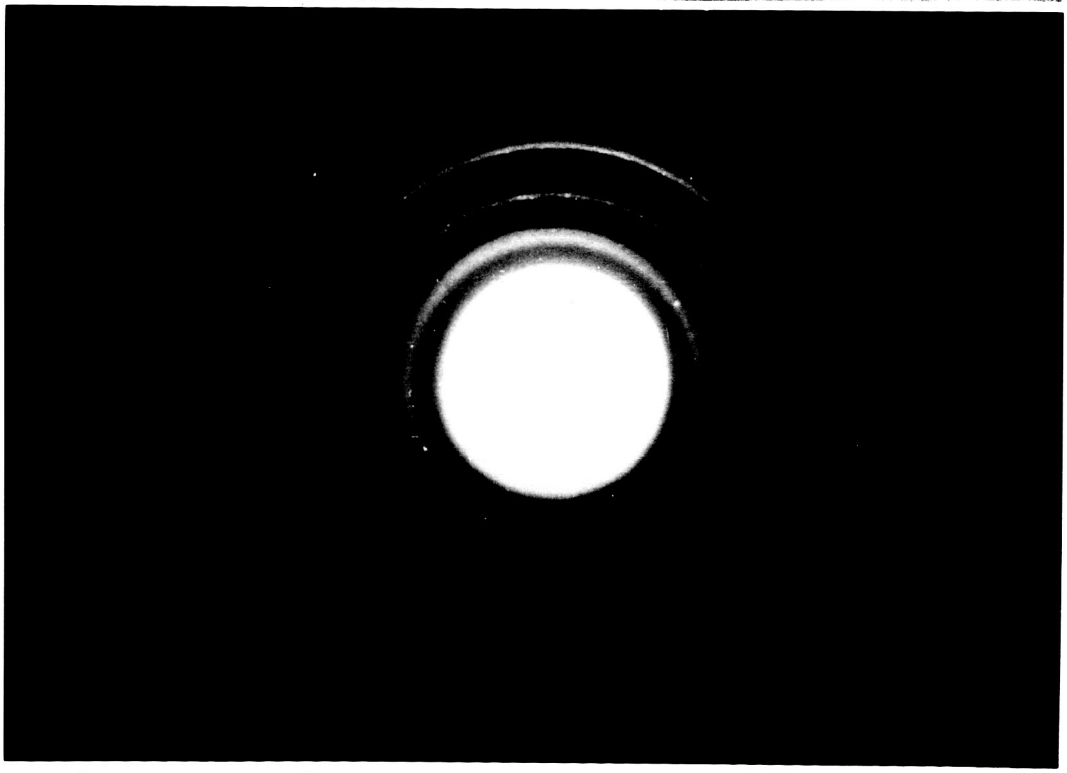


[illegible] : [illegible] ELECTRON DIFFRACTION PATTERN OBTAINED [illegible] ON THIN CARBON-FORMVAR FILM [illegible] EXPOSED DURING THE [illegible] COLLECT MICROMETEOROID DEBRIS DURING THE PEAK OF THE LEONI[illegible] SHOWER. ("LUSTER" SERIES No. [illegible]).

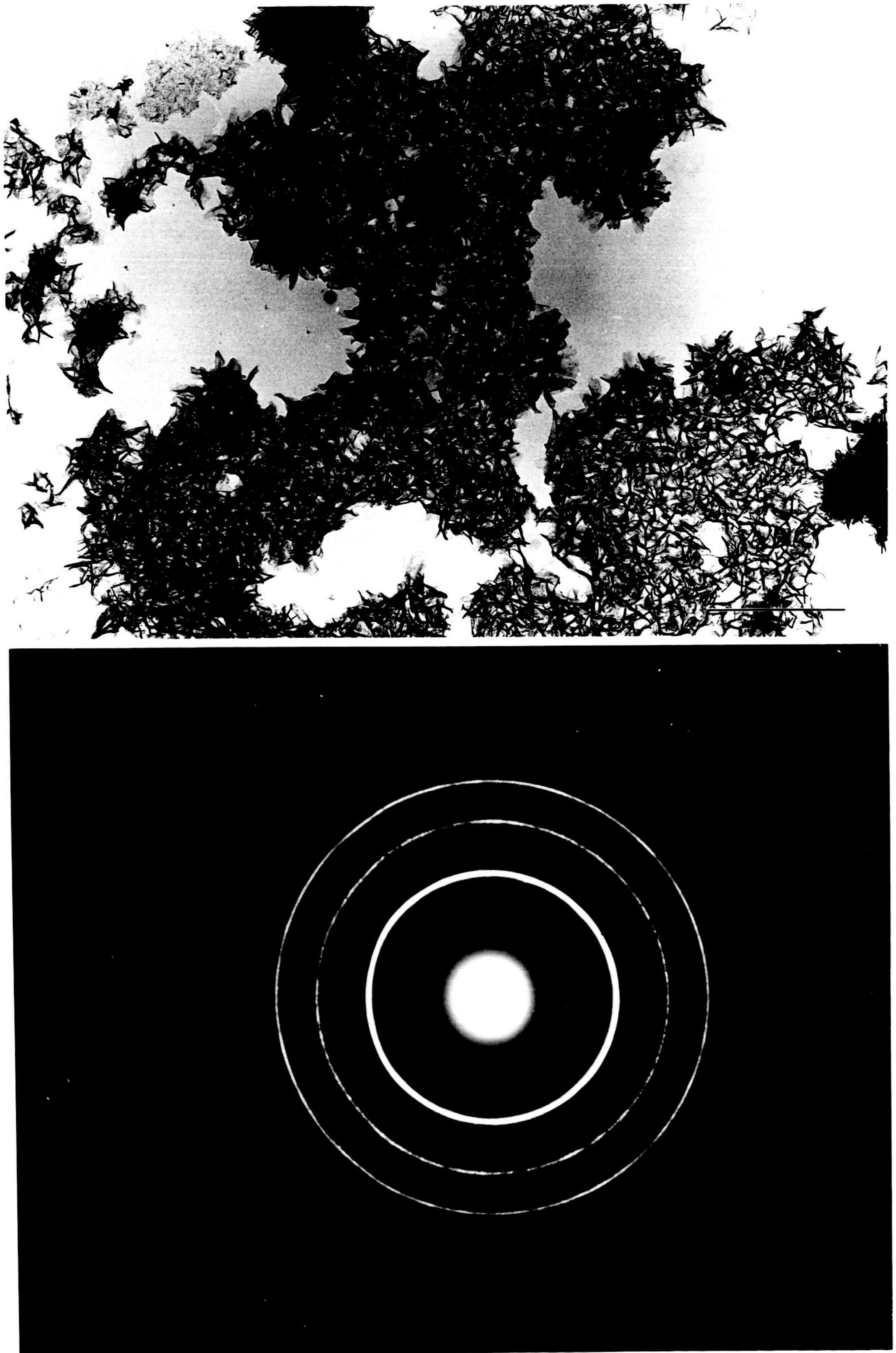
SPHERICAL [illegible] PARTICLES OF THIS SIZE (ca. 1μ diameter) WERE RARELY ENCOUNTERED. UNDER ELECTRON BOMBARDMENT OF MODERATE INTENSITY PART OF THE MATERIAL EVAPORATES AND RECONDENSES AROUND THE LARGE PARTICLE FORMING COLLECTIONS OF MINUTE SPHERICAL OR POLYGONAL PARTICLES (ca. 100 to 2000 Å diameter). ELECTRON DIFFRACTION PATTERNS RECORDED FROM THESE AREAS EXHIBIT CHARACTERISTIC SHARP RING PATTERNS WITH SPACINGS WHICH ARE VERY SIMILAR TO THOSE OBTAINED FROM CANYON DIABLO METEORITE PARTICLES (see FIG. M-1),AND ALSO TO THE DIFFRACTION PATTERNS OBTAINED FROM MICROMETEORITE PARTICLES DESCRIBED BY HEMENWAY & SOBERMAN (Astron. Journal, 67,256,1962;Figs.22,25).THEY MAY CORRESPOND MAINLY TO THE Fe-Ni alloys (taenite and kamacite).IN ADDITION THERE ARE CHARACTERISTIC SMALL-ANGLE PATTERNS WITH SPACINGS OF 12-17Å WHICH ARE BEING FURTHER INVESTIGATED. MAGNIFICATION:(Original mag.:15,300 X).DIFFRACTION PATTERN RECORDED AT 75 kV.







[illegible]-2 : ELECTRON MICROGRAPH AND SELECTED-AREA ELECTRON DIFFRACTION PATTERN OF PARTICLES FROM [illegible] METEORITE AFTER [illegible] FOR A FISCHER-TROPSCH TYPE REACTION BETWEEN CARBON MONOXIDE AND HYDROGEN. [illegible] OF THE ENRICO FERMI INSTITUTE FOR NUCLEAR STUDIES, AND BY DR. M. STUDIER OF ARGONNE NATIONAL LABORATORY, [illegible]. [illegible] METEORITE POWDER WAS HEATED WITH C-H-O MIXTURES IN A QUARTZ TUBE AT [illegible]°-[illegible]°C, YIELDING ALIPHATIC AND AROMATIC HYDROCARBONS. [illegible] QUANTITIES OF THE REACTED METEORITE POWDER WERE PREPARED ACCORDING TO THE SURFACE-FILM TECHNIQUE ON A CLEAN WATER SURFACE, AND [illegible] PICKED UP ON ULTRATHIN ([illegible]) CARBON FILMS FOR ELECTRON MICROSCOPY. IN ADDITION TO AREAS CONTAINING METALLIC PARTICLES, [illegible] PORTIONS OF THE SPECIMEN FEATURE LIGHT (CRYSTALLINE) LAYERS COATED WITH AN AMORPHOUS MATERIAL WHICH VOLATILIZES UNDER THE [illegible] BEAM (top frame). SELECTED-AREA DIFFRACTION PATTERNS FROM SUCH AREAS (below) EXHIBIT PATTERNS OF SHARP RINGS, AND TYPICAL CRYSTALLINE PATTERNS WHICH ARE BEING FURTHER INVESTIGATED. MAGNIFICATION: (Original, top: 10[illegible],[illegible]X; after reproduction: [illegible],000 X).



[illegible]: ELECTRON MICROGRAPH A[illegible] SELECTED-AREA ELECTRON DIFFRACTION PATTERN OF MA[illegible] [illegible] [illegible]ATING AS [illegible] [illegible]. [illegible] CANYON DIABLO METEORITE POWDER WAS TREATED WITH [illegible]-H-O MIXTURES IN A [illegible], [illegible] [illegible] QUANTITIES OF THE REACTED METEORITE POWDER WERE THEN PREPARED IN THE LABORATORY [illegible] [illegible] WATER SURFACE, AND THE [illegible] [illegible] ON [illegible] (ca. [illegible] A) CARBON FILMS FOR ELECTRON MICROSCOPY. [illegible] [illegible] OF FINE FILAMENTOUS METALLIC PARTICLES (whiskers) [illegible] WITH [illegible] [illegible] [illegible] [illegible] DIFFRACTION PATTERN RECORDED [illegible] WITH [illegible] ILLUMINATION.

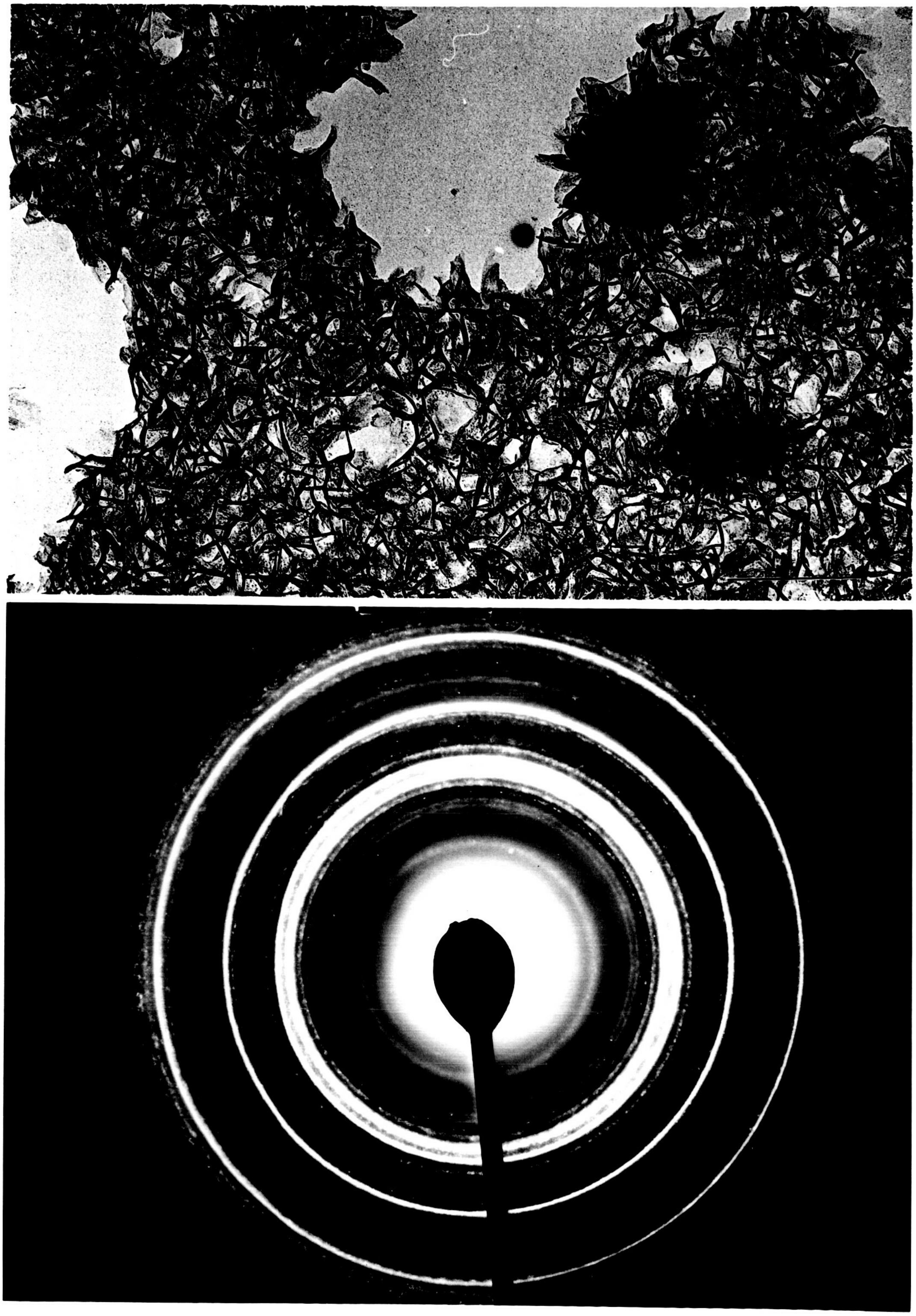
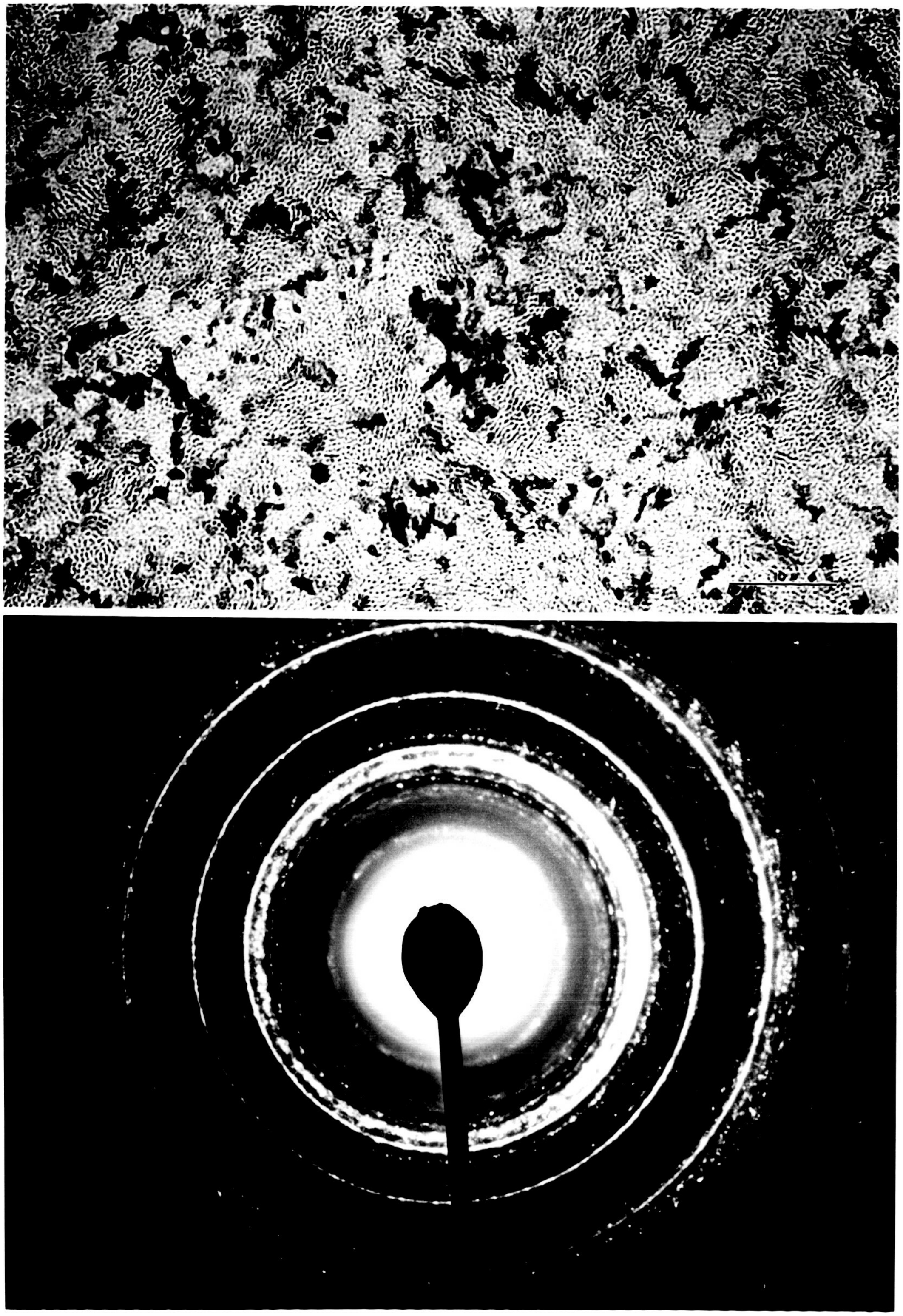


FIGURE M-4: ELECTRON MICROGRAPH AND SELECTED-AREA ELECTRON DIFFRACTION PATTERN OF MATERIAL FROM CANYON DIABLO METEORITE PARTICLES AFTER REACTING AS CATALYSTS FOR A FISCHER-TROPSCH TYPE REACTION BETWEEN CARBON MONOXIDE AND HYDROGEN.

SAME SPECIMEN AS IN FIGURE M-3 SHOWN AT HIGHER MAGNIFICATION (top). HIGH RESOLUTION SELECTED-AREA DIFFRACTION PATTERN WITH CENTRAL BEAM STOP. THESE EXPERIMENTS ILLUSTRATE THE UNIQUE ADVANTAGES OF CORRELATED ELECTRON MICROSCOPY AND ELECTRON DIFFRACTION STUDIES FOR DIRECT VISUALIZATION OF THE REACTION PRODUCTS ON SUBMICROSCOPIC METEORITE PARTICLES UNDER CONTROLLED CONDITIONS.

Original magnification: 100,000 X.



[illegible] M-[illegible]: HIGH RESOLUTION ELECTRON MICROGRAPH AND SELECTED-AREA ELECTRON DIFFRACTION PATTERN OF EVAPORATED THIN FILM OF CANYON DIABLO METEORITE AS CONTROL FOR REACTED SPECIMENS SHOWN IN FIGURES M- to M-. IN THESE EXPERIMENTS, CARRIED OUT IN COLLABORATION WITH DR. E. ANDERS, DR. M. [illegible] OF THE ENRICO FERMI INSTITUTE FOR NUCLEAR STUDIES, AND DR. [illegible] OF ARGONNE NATIONAL LABORATORY, [illegible] CANYON DIABLO METEORITE POWDER WAS FIRST EVAPORATED IN [illegible] VACUUM ([illegible] mm Hg) [illegible] ELECTRON MICROSCOPE STUDIES. [illegible] FILM WAS [illegible] [illegible] [illegible] DIFFRACTION PATTERN [illegible] material. Original magnification: [illegible].

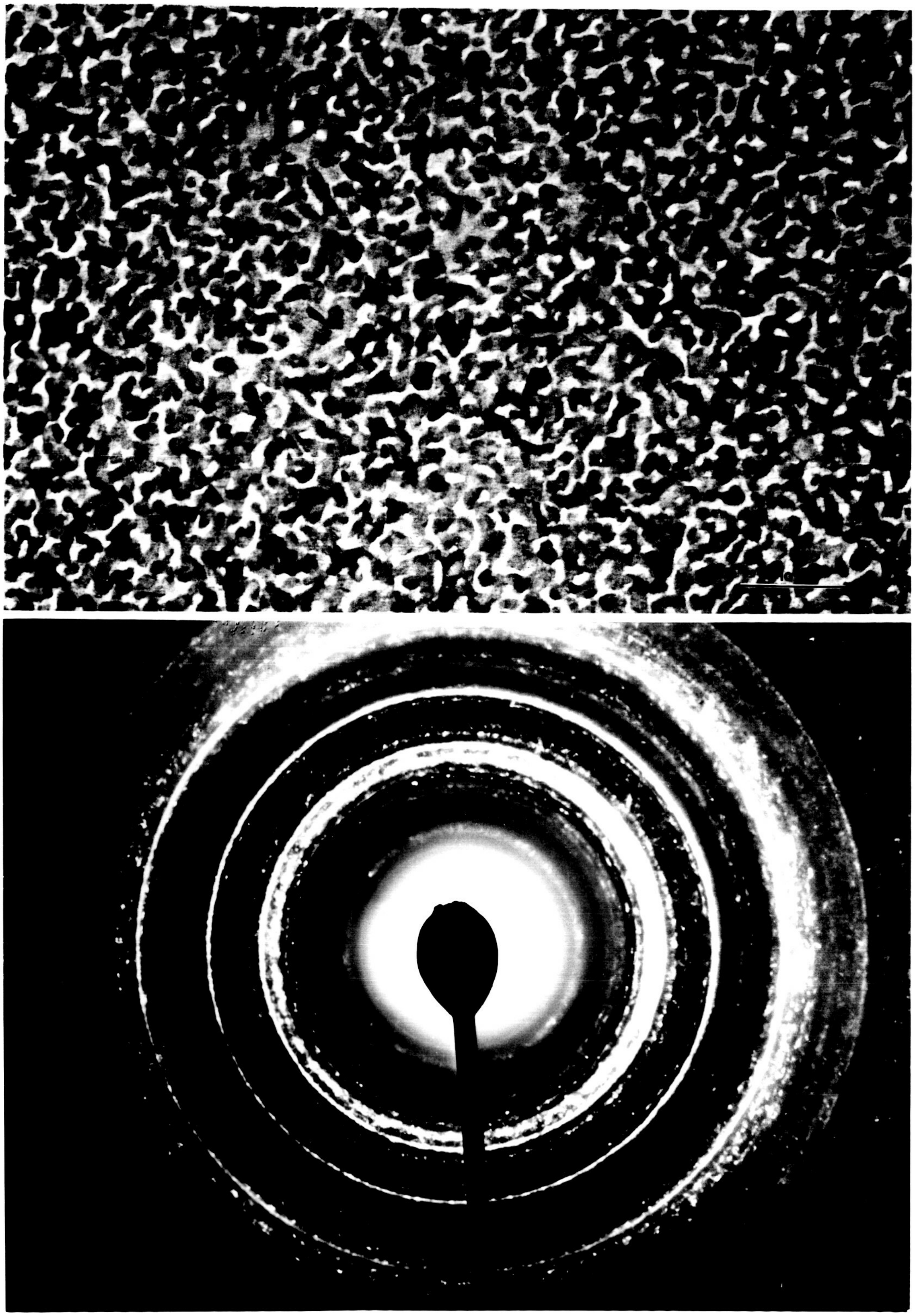


FIGURE M-6: HIGH RESOLUTION ELECTRON MICROGRAPH AND SELECTED-AREA ELECTRON DIFFRACTION PATTERN OF EVAPORATED THIN FILM OF CANYON DIABLO METEORITE ACTING AS CATALYST FOR A FISCHER-TROPSCH TYPE REACTION BETWEEN CARBON MONOXIDE AND HYDROGEN. EXPERIMENTAL CONDITIONS AS DESCRIBED IN FIGURE M-5. NOTICE APPEARANCE OF TYPICAL DEPOSITS OF (ORGANIC) MATERIAL ON THE POLYCRYSTALLINE THIN (Ni-Fe) FILM SHOWN IN FIG. M-5. CHARACTERISTIC HIGH RESOLUTION, SELECTED-AREA ELECTRON DIFFRACTION PATTERNS RECORDED FROM THESE REACTED AREAS (below) ARE BEING FURTHER INVESTIGATED IN ATTEMPT TO IDENTIFY SOME OF THE CHARACTERISTIC ALIPHATIC AND AROMATIC HYDROCARBONS KNOWN TO RESULT FROM THIS REACTION. Original magnification: 400,000 X.

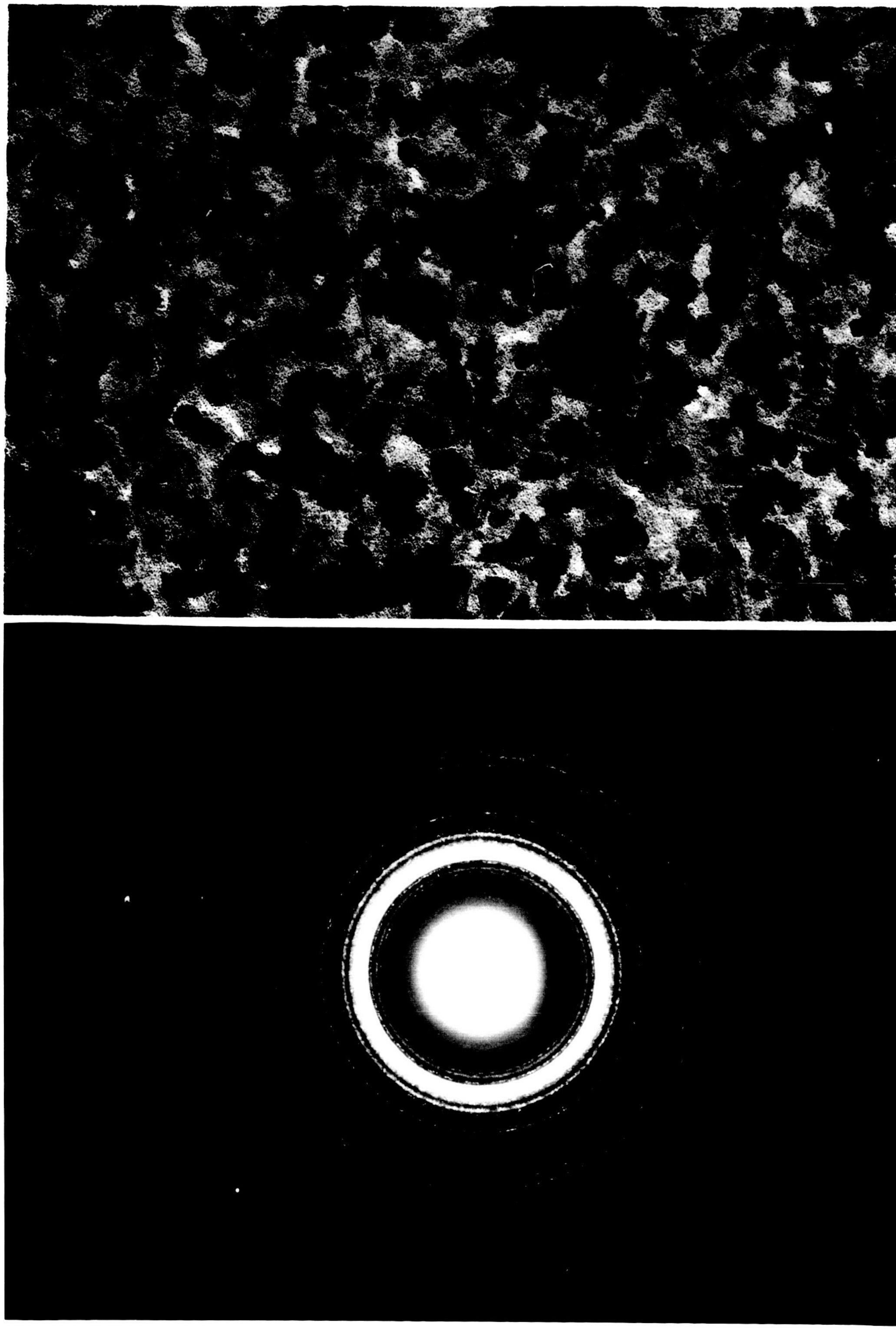


FIGURE M-7: HIGH RESOLUTION ELECTRON MICROGRAPH AND SELECTED-AREA ELECTRON DIFFRACTION PATTERN OF EVAPORATED THIN FILM OF CANYON DIABLO METEORITE AFTER REACTING AS CATALYST FOR A FISCHER-TROPSCH TYPE REACTION BETWEEN CARBON MONOXIDE AND HYDROGEN. EXPERIMENTAL CONDITIONS AS DESCRIBED IN FIGURES M-5,6. SELECTED AREA ELECTRON DIFFRACTION PATTERN(below)RECORDED WITHOUT BEAM STOP. Original magnification: 800,000 X.

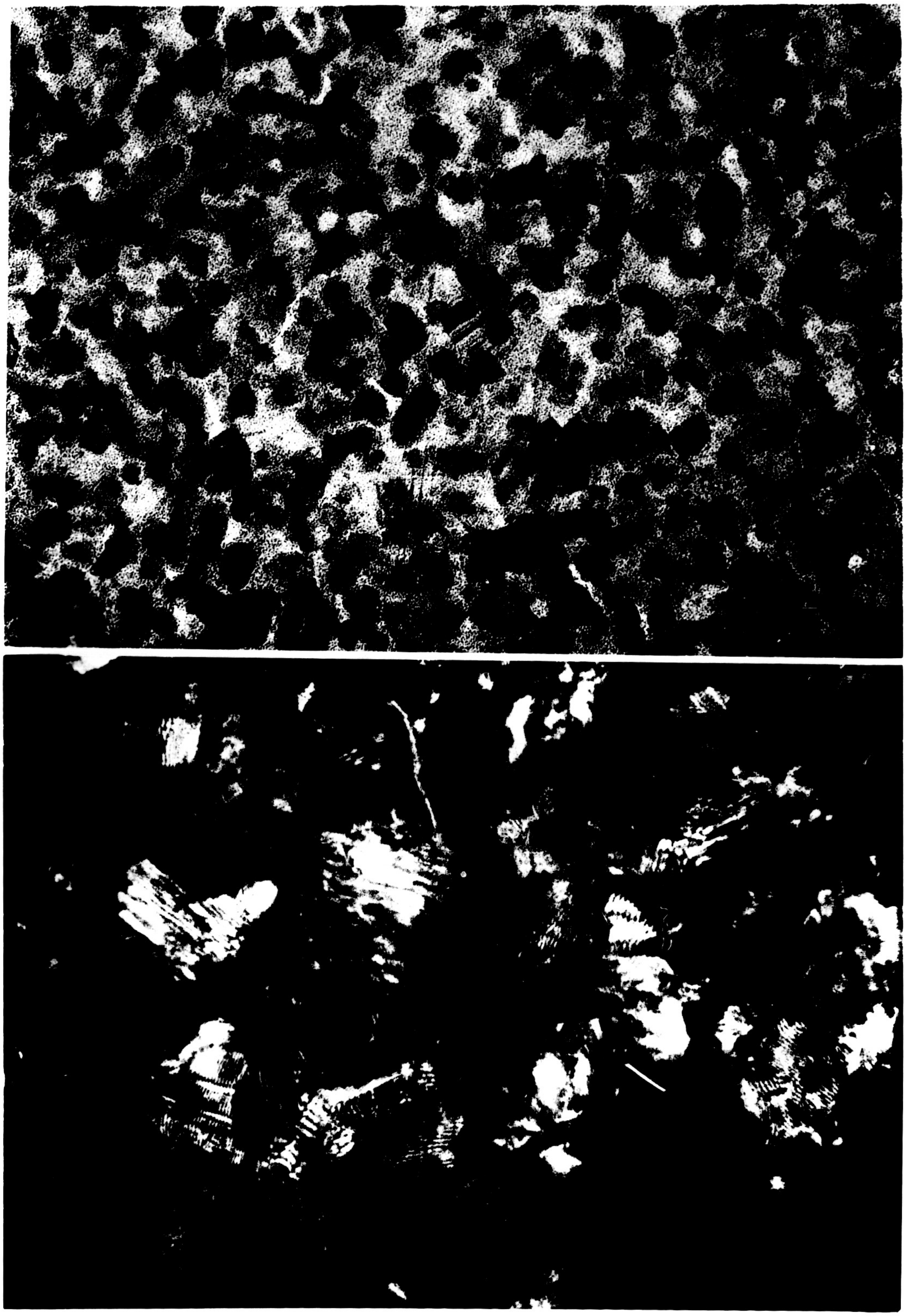


FIGURE M-[illegible]: HIGH RESOLUTION BRIGHT-FIELD AND DARK-FIELD ELECTRON MICROGRAPHS OF EVAPORATED THIN FILM OF CANYON DIABLO METEORITE [illegible] AS CATALYST FOR A FISCHER-TROPSCH TYPE REACTION BETWEEN CARBON MONOXIDE AND HYDROGEN. EXPERIMENTAL CONDITIONS A[illegible] [illegible] M-[illegible] to 7. THE CHARACTERISTIC HIGH RESOLUTION DARK-FIELD MICROGRAPHS RECORDED WITH OBLIQUE MICROBEAM ILLUMINA[illegible] [illegible] TO YIELD IMPORTANT ADDITIONAL INFORMATION ON THE ORGANIC COMPONENTS OF FILM. Original magnification: [illegible], [illegible].